Commission of the
European Communities
Joint Research Centre

Petten Establishment

Annual Report 1977

COMMISSION OF THE EUROPEAN COMMUNITIES

JOINT RESEARCH CENTRE

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INTRODUCTION

1977 marked the beginning of a new multiannual research programme for the European Commission's Joint Research Centre. The Petten Establishment's scientific programmes were begun in 1975 in a revision of the previous four year's exercise and consequently the two programmes which concern high temperature materials and organic products have been extended into the 1977-1980 period without significant alteration.

Regarding the exploitation of HFR (High Flux Reactor), the year was one of steady, on-schedule operation with high utilization, although this fell off slightly in the autumn due to overloading of our capsule project engineering team and manufacturing services. We are pleased to note that the HFR Users' Meeting, held in October, drew about 100 participants from Europe and America and demonstrated a lively interest in the Commission's materials testing reactor. Technical improvements to the plant are being examined as one means of maintaining or even increasing this interest in the 1980's.

Following the wishes of the Council of Ministers, new Advisory Committees for Programme Management have been set up for all the Joint Research Centre's activities and those for HFR and High Temperature Materials Programmes have met at Petten. This latter Committee is entirely new to its task, the programme having been served until 1977 by a number of ad hoc meetings of national experts. The work of the Organic Chemistry Laboratory falls under the wings of the Community Reference Bureau Advisory Committee, who have met in Brussels and strongly encouraged the development of the activity.

As in other years, the satisfactory results could not have been achieved without the hard work of our staff. I hope something of this can be seen between these covers.

P.J. van Westen
Director

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I HFR DIVISION

INTRODUCTION

During the first year of the current programme of the Joint Research Centre, operation and utilization of the Commission's materials testing reactor HFR Petten continued as scheduled. The reactor was operated over 282 days at 45 MW, corresponding to 79 $^{o}/o$ availability. The scheduled long shut-downs in spring and summer have been used for thorough inspection and maintenance. The tight inspection and maintenance schedule, together with a continuous process of replacement and renewal of components, has kept the plant in an excellent condition. Outages due to component failures are practically unknown.

The reactor can be considered as a large testing facility for material specimens and components of operating and future nuclear power plants. Its main use is therefore directed towards the support of R & D programmes of European Research Centres, nuclear industry, and own JRC projects. Furthermore experiments in solid state and nuclear physics are carried out and radioisotopes are produced. In terms of the nature of specimens irradiated, the distribution in 1977 was approximately as follows (in percentages of the used capacity):

Graphite, and HTR fuel	19
Structural materials	25
LWR fuel	13
Fast breeder reactor fuel	5
Neutron physics, and radioisotopes	30
Miscellaneous	8

After the dismantling of the high pressure water loop, only capsule-type experiments are carried out in HFR. Special devices have been developed for various reactor safety related experiments, for in-pile creep measurements, in-pile instrumentation development, fracture mechanics studies etc. Close collaboration between all organization and covering all specialities involved in reactor materials research has turned out to be the most efficient approach for irradiation testing.

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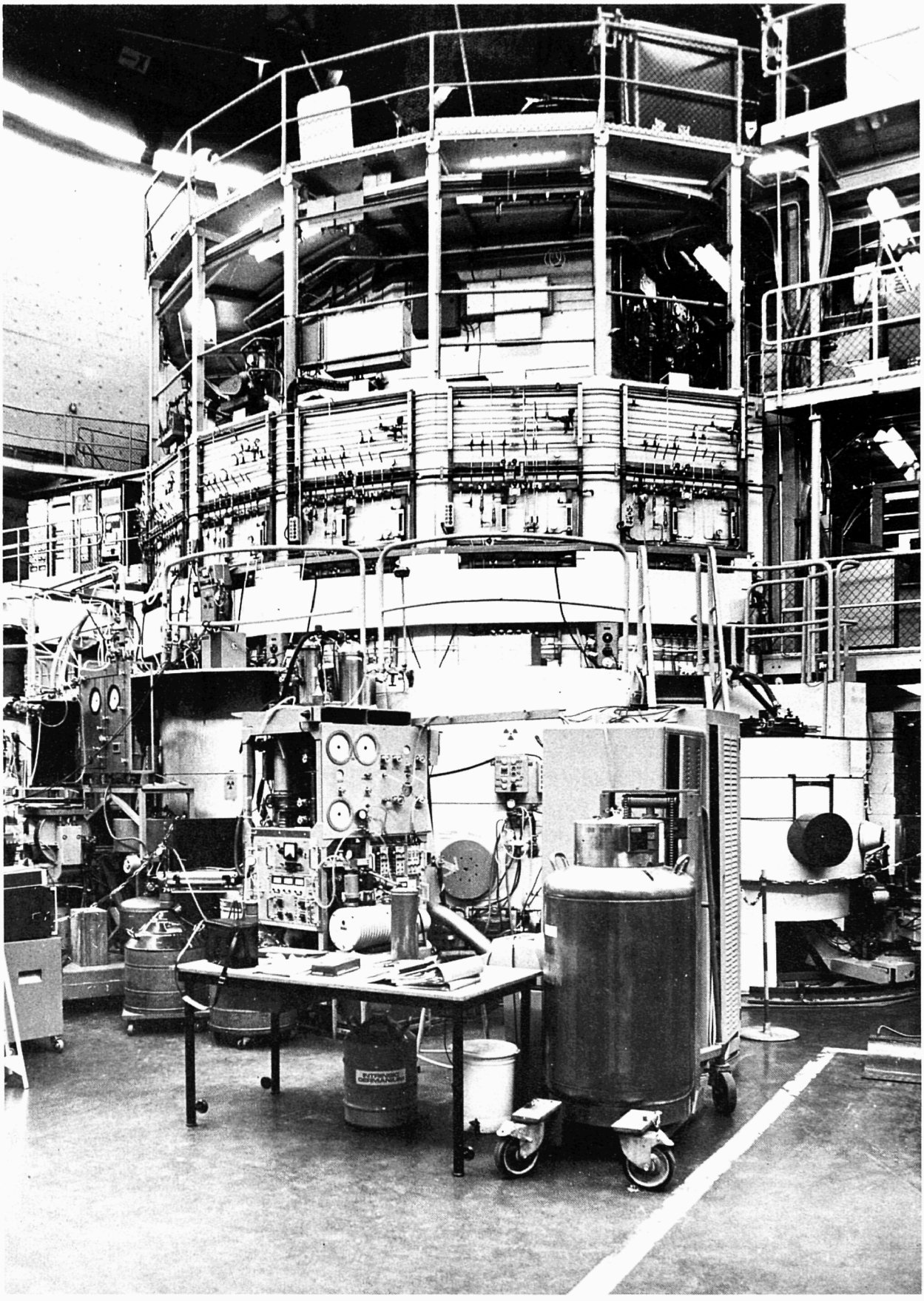

Fig. 1 HFR Petten. Reactor Hall.

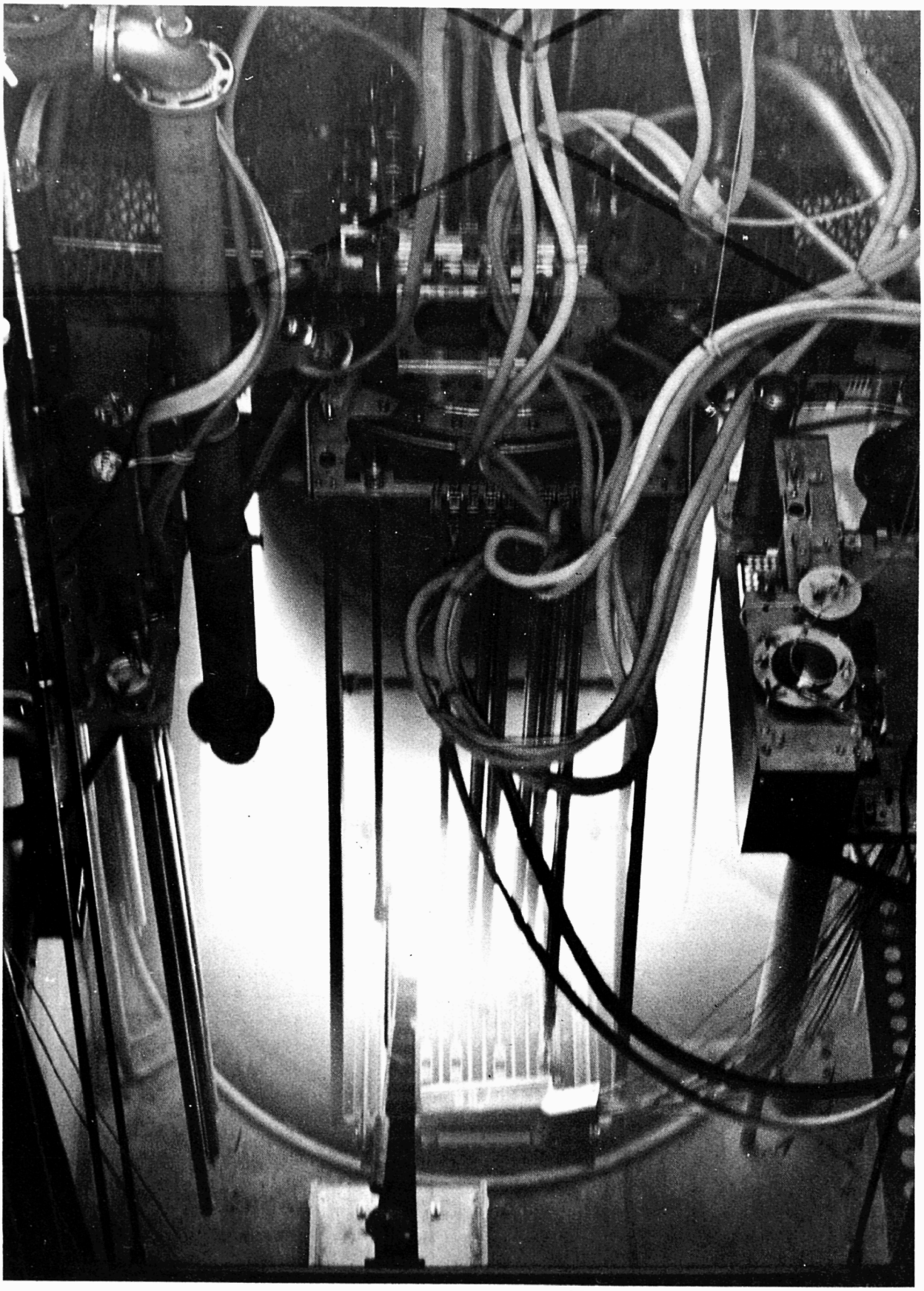

Fig. 2 HFR Petten. Reactor Pool.

1 OPERATION OF HFR PETTEN

1.1 GENERAL

Typical data of the materials testing reactor HFR Petten *) **) are resumed in Table 1. The data of Table 1 refer to the core configuration used since April 1977.

Table 1: Summary of nuclear and thermal properties.

reactor power	45 MW	
number of fuel assemblies	33	
number of control members	6	
number of in core irradiation positions	9	
number of reflector irradiation positions	8	
number of horizontal beam tubes	11	
number of pool side facility positions	12	fresh fuel loading
fuel charge of fresh fuel assemblies	390 g ^{235}U	of the fissile
boron charge in the side plates of fresh fuel assemblies	1000 mg ^{10}B	control member
total fuel charge	11 kg ^{235}U	followers: 270g
volume of core	0.2 m^3	
average thermal flux density in inner fuel position	1.5 x 10^{18} $m^{-2}s^{-1}$	
maximum thermal flux density in inner fuel position	2.1 x 10^{18} $m^{-2}s^{-1}$	
maximum fast flux density in in core experim. position	2.9 x 10^{18} $m^{-2}s^{-1}$	equivalent fission
max. thermal flux density in in core experim. position	2.0 x 10^{18} $m^{-2}s^{-1}$	flux density
typical nuclear heating in graphite: in core positions	13 W/g	
reflector positions	6.1 W/g	maxima, small
pool-side facility	4 W/g	samples
flow rate of primary coolant	4300 m^3/h	
coolant speed in fuel assembly	7.1 m/s	
coolant speed in filler element	0.2 to 7.1 m/s	
inlet temperature of coolant	313 K (40°C)	
outlet temperature of coolant	323 K (50°C)	
temperature difference across the reactor core	10 K	
average heat flux density in mid position	1.00 MW/m^2(100 W/cm^2)	
maximum heat flux density in mid position	1.60 MW/m^2(160 W/cm^2)	
absolute pressure above the reactor core	340 kN/m^2 (3.4 bar)	
pressure difference over the reactor core	110 kN/m^2 (1.1 bar)	

1.2 HFR OPERATION

1.2.1 Operation Summary

The operating pattern of the HFR follows a 28-days operation period, comprising 26 days of actual reactor operation, followed by a 2-days regular reactor shut-down. This shut-down period is used for installation and reloading of the irradiation rigs, maintenance to the reactor and experiment facilities, and for refuelling.

*) *HFR Annual Report 1976.*

**) *H. Röttger et al.: "High flux materials testing reactor HFR Petten. Characteristics of facilities and standard irradiation devices (Edition 1977-1978)" EUR 5700e (October 1977).*

The 26 days cycle duration has been realized by a fuel loading in five zones and a maximum burn-up of 55%. The fresh assemblies containing 390 g ^{235}U are loaded in the zone between periphery and centre, the nearly spent assemblies in the central zone, and other assemblies are placed in the peripheral zones of the core. By moving the fuel assemblies stepwise from one zone to another after each reactor cycle, stable irradiation conditions can be maintained for successive cycles.

Two changes to the reactor core configuration have been introduced in 1977, viz.

- creation of a new in-core high flux irradiation position (G5), after unloading of the high pressure water loop in pile section
- application of the so-called semi-peripheral fuel element loading mode resulting in flux increases in isotope positions D2/D8 - F2/F8, as well as in an improved thermal safety of the reactor.

Fig. 3 shows the 1977/78 configuration, with typical neutron flux and nuclear heating figures.

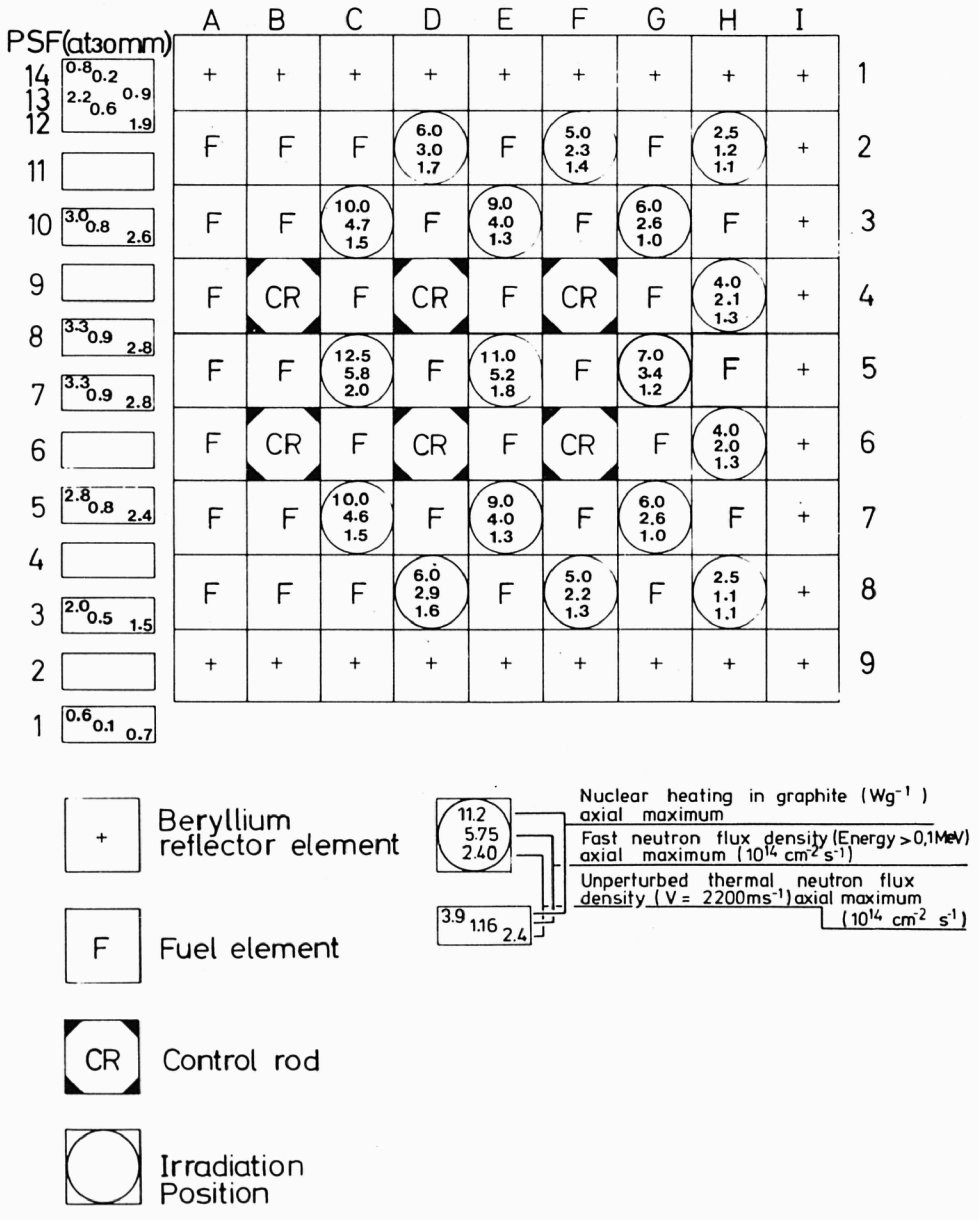

Fig. 3 *Materials testing reactor HFR. 1977/78 core configuration and nuclear characteristics of the irradiation positions.*

Table 2 contains the annual operating calendar. It largely corresponds to the schedule set up by the end of 1976, with the exception of the last week in December: the annual maintenance period 1978 has been shifted from March to January, it actually started on December 26, 1977. Table 3 resumes overall operating figures, together with the number of perturbations by reactor scrams and power decreases during the cycles. In total, 17 perturbances were caused by experiments, and 14 by reactor systems. As in previous years, the overall loss of operating time remained small.

Table 2 : HFR 1977. Operating history.

HFR Cycle Nr.	Main dates (1977) Begin	End	Start	Operating time (days) below 45 MW	at 45 MW	Availability (%)	Total energy production (MWd)	Core configuration*), remarks
77.01	01.01	24.01	01.01	0.1	22.4	93.9	1014	HPL
77.02	25.01	21.02	26.01	0.2	26.1	93.9	1183	HPL
77.03	22.02	11.04	11.03	0.3	30.4	63.5	1401	incl. maintenance
77.04	12.04	09.05	13.04	0.2	25.9	92.8	1169	
77.05	10.05	06.06	12.05	0.6	25.1	90.5	1140	
77.06	07.06	04.07	08.06	0.2	25.8	92.6	1167	
77.07	05.07	05.09	10.08	0.4	26.2	44.2	1254	incl. holidays
77.08	06.09	03.10	07.09	0.7	24.5	89.0	1121	
77.09	04.10	31.10	05.10	0.1	26.2	96.0	1209	SPFL
77.10	01.11	28.11	02.11	0.3	25.9	93.0	1172	SPFL
77.11	29.11	31.12	30.11	0.1	23.7	72.2	1072	incl. first part of maintenance
				3.2	282.2	average 78.6	12.902	

*) *core configuration codes: HPL with high pressure loop loaded*
SPFL semi-peripheral fuel loading core

Table 3 : Operating Summary

		First half 1977	Second half 1977
Integrated reactor power	MWd	6897	6005
Average reactor power	MW	45.1	45.5
Operating time	%	84.5	72
Unscheduled shutdown-time	%	1.4	1.1
Unscheduled shut-downs	--	13	9
Unscheduled power decreases	--	--	3
Planned scrams	--	2	2
Planned power decreases	--	1	1
Fuel consumption	gr ^{235}U	8614	7600
Release through stack (^{41}Ar)	Ci	234	231

1.2.2 Maintenance Summary

Inspection and servicing of all major components and systems of the plant have taken place
- during the February/March maintenance outage,
- during the last week of the summer holiday period,
- during the last days of December.

On the other hand maintenance activities during routine inter-cycle shut-downs have been kept to the strict minimum resulting in typical outage durations of no more than 40 hours for refuelling and experiment unloading/loading.

Some of the maintenance operations are briefly listed as follows:
- inspection and overhauling of the control rod drive mechanisms (this was carried out in the summer outage rather than during the spring maintenance period, as a consequence of high gamma fields near the reactor bottom plate caused by lost ^{192}Ir pellets),
- clean-up of aluminium pipes on the heat exchanger secondary side,
- overhauling of all four secondary pumps,
- improvement of ion exchanger resin replacement techniques,
- dimensional measurements on core box structures, lower grid holes and clamp-down (grid) bars,
- PSF core box wall video recording,
- introduction of new wide range neutron flux measuring channels,
- two reactor containment building leak tests,
- routine maintenance and repair work on numerous other systems.

The overall good technical condition of the plant has been confirmed by these activities.

1.2.3 Improvements, Support, Development

Buildings

An inner area according to the IAEA recommendations on physical protection of nuclear material had been defined in 1976. During the reporting period the security measures have been implemented, both by modifications of the existing building and by detecting instrumentation. Detailed drawings have been elaborated for an extension of the reactor building complex.

New Reactor Vessel

A preliminary feasibility study has been carried out for the replacement of the present reactor vessel (Fig. 4). The reasons for studying a new vessel are:

1. a back-up has to be available should licensing problems arise, after 1980, with the present vessel
2. the experience gathered in nearly 20 years of utilization is used for improving
   - the thermohydraulics of the system,
   - the irradiation facilities.

Presently it is intended to design and calculate the new vessel in 1978 and to manufacture in 1979/80. The earliest possible date for replacement would therefore be early in 1981.

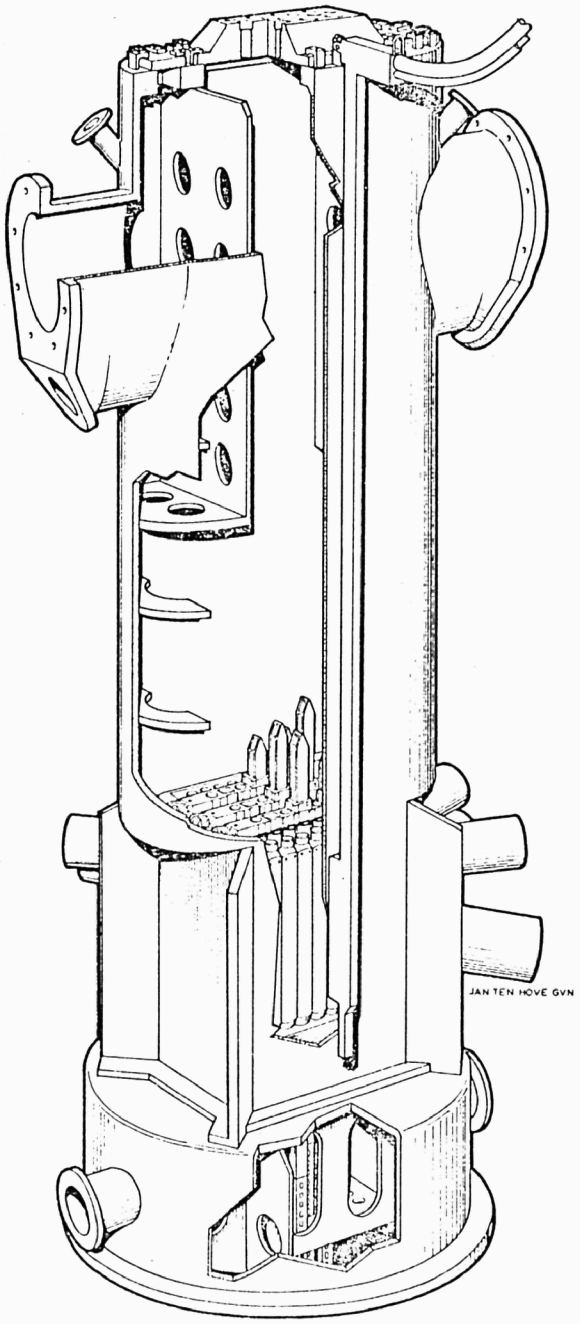

Fig. 4 Present proposal of future HFR vessel.

1.2.4 Development of Neutron Spectrum Determinations

Cross section libraries

A comparison has been made of cross section libraries from various origins. The new cross section library, coded DOSCROSS77, is available in the 620 groups SAND-II format. The library comprises 49 detector cross section sets and 3 cover cross section sets. The basic data for this library were obtained from the ENDF/B-IV dosimetry file. The contents of this new cross section library have been made available in a report*).

A damage cross section library, coded DAMSIG77, was prepared. This library contains:

a) the data of the library SANDAMAGE;
b) the data present in the recommendations of the Euratom Working Group on Reactor Dosimetry and
c) the damage cross sections for vanadium and niobium.

These damage cross sections are available in the 620 groups SAND-II format. The materials available in the library are graphite, stainless steel, aluminium, silicium, chromium, iron, nickel, copper, zirconium, molybdenum, tungsten, vanadium and niobium. Also some activation cross sections are incorporated in the library so that all information is present to calculate the damage to activation ratio (DAR) for a spectrum of interest.
The contents of this damage cross section library have been described in a report**).

Attenuation by foil covers

Corrections have to be applied for the shielding by covers (cadmium or boron) which are used for detector sets in neutron spectrum determinations. The covers are used

*) *W.L. Zijp, H.J. Nolthenius, and N.J.C.M. van der Borg: Cross section library DOSCROSS77 (in the SAND-II format)., ECN-25 (Netherlands Energy Research Foundation ECN, Petten), August 1977.*
**) *W.L. Zijp, K.H. Appelman, and H.J. Nolthenius, Damage cross section library (DAMSIG-77), ECN-36, February 1978.*

to shift the detector response to higher energies. The attenuation calculation is rather complicated due to the complex geometry which is present for an actual detector set in an isotropic neutron field. For this reason a programme (COVER) has been written in which 5 different simplified models for the attenuation can be used. In this way data can be obtained on the influence of parameters and models on the calculated attenuation. The work on this topic has not yet been finished.

The certainty parameter in unfolding

In neutron spectrum unfolding procedures one has a set of equations of the following type

$$a_i = \int_{\phi}^{\infty} \sigma_i(E).\,\phi_E(E)\,.\,dE \qquad \text{for } i = 1, 2....n).$$

The quality of the information which is applied in the unfolding can be described with the following expression

$$\gamma_j = -\frac{ds}{d\phi_j} \Big/ \frac{s}{\phi_j} \,,$$

where s is the sum of the squares of the weighted relative deviation between measured and calculated reaction rates in energy group j, and ϕ_j is the group flux density.
Some experience on the application of this expression to calculate the certainty parameter has been presented at an IAEA specialist meeting *).

The modifying factor in unfolding

The modification that is applied in SAND-II on the input spectrum is determined by a set of activation reactions. In order to study the influence of particular reactions from this set a small program has been written that plots the modification contribution of each reaction of the detector set. Some results obtained with this programme are shown in Fig. 6.
More details on this modifying parameter are given in a report which has been presented at an IAEA specialist meeting **).

Comparison of neutron spectrum unfolding codes

Three computer programs (CRYSTAL BALL, RFSPJÜL and SAND-II) for neutron spectrum unfolding have been compared. In this comparison three different fast neutron spectra have been applied (CFRMF, ΣΣ and STEK) and two different cross section libraries were used. In comparison it was found that the three unfolding programs give comparable results for the three spectra if the modifications are small. When appreciable modifications have to be applied, the three unfolding codes show different behaviour, especially in the region between 10^{-3} and 1 MeV where the activation detectors give a very poor response.

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*) *W.L. Zijp, and H.Ch Rieffe: The certainty parameter in unfolding. ECN-77-148, November 1977.*

**) *W.L. Zijp and H.Ch. Rieffe: The modifying factor in unfolding. ECN-77-158, November 1977.*

In Fig. 5 the improvement ratio and the characteristic modification of the three codes for a very simple input activity set (2 values) are shown.
The results of the comparison were presented at the 2nd ASTM-Euratom Symposium on Reactor Dosimetry *).

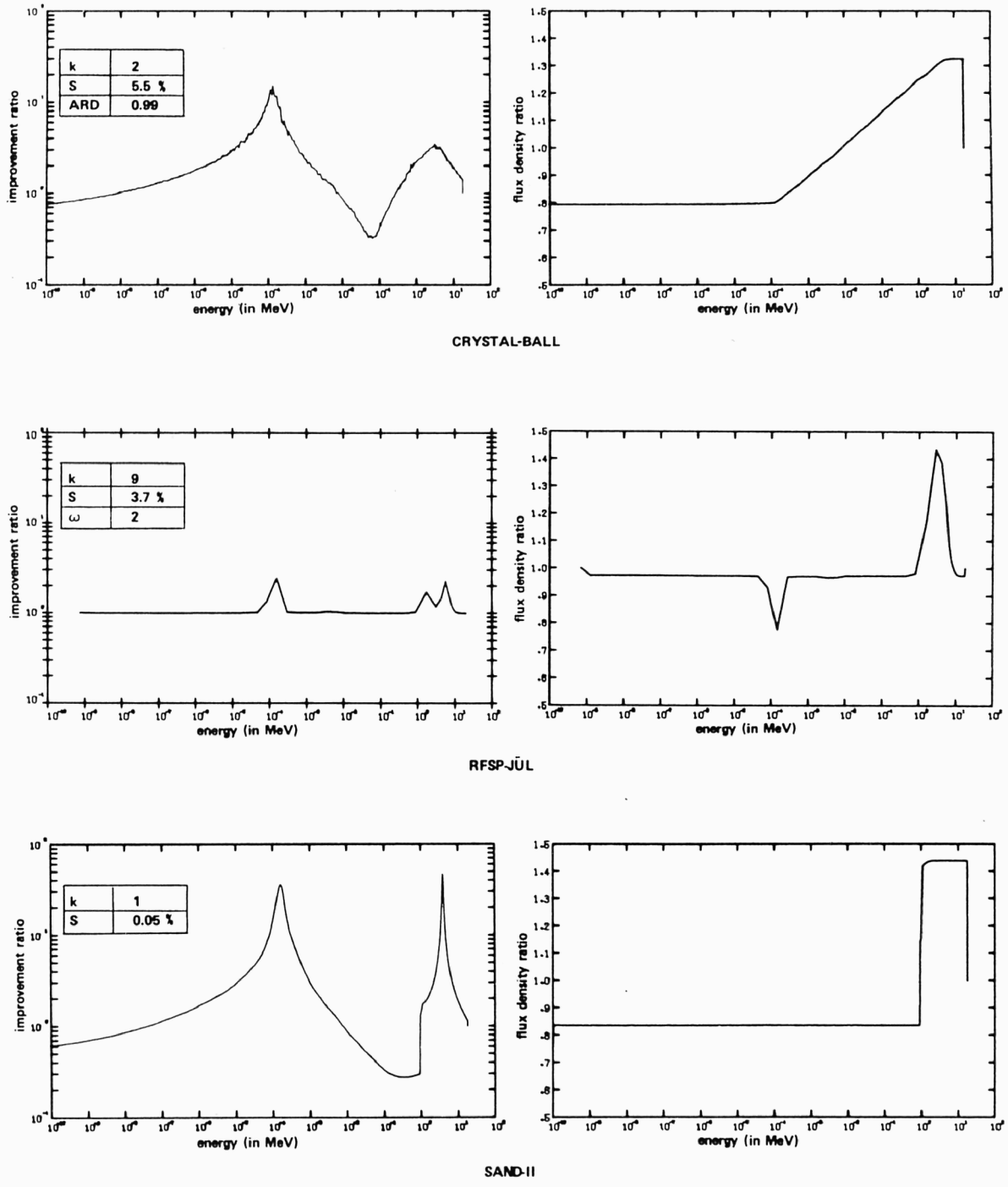


Fig. 5 *Results for the Co and Ni set.*

*) *W.L. Zijp and H.J. Nolthenius: Comparison of neutron spectrum unfolding codes. ECN-77-111, September 1977.*

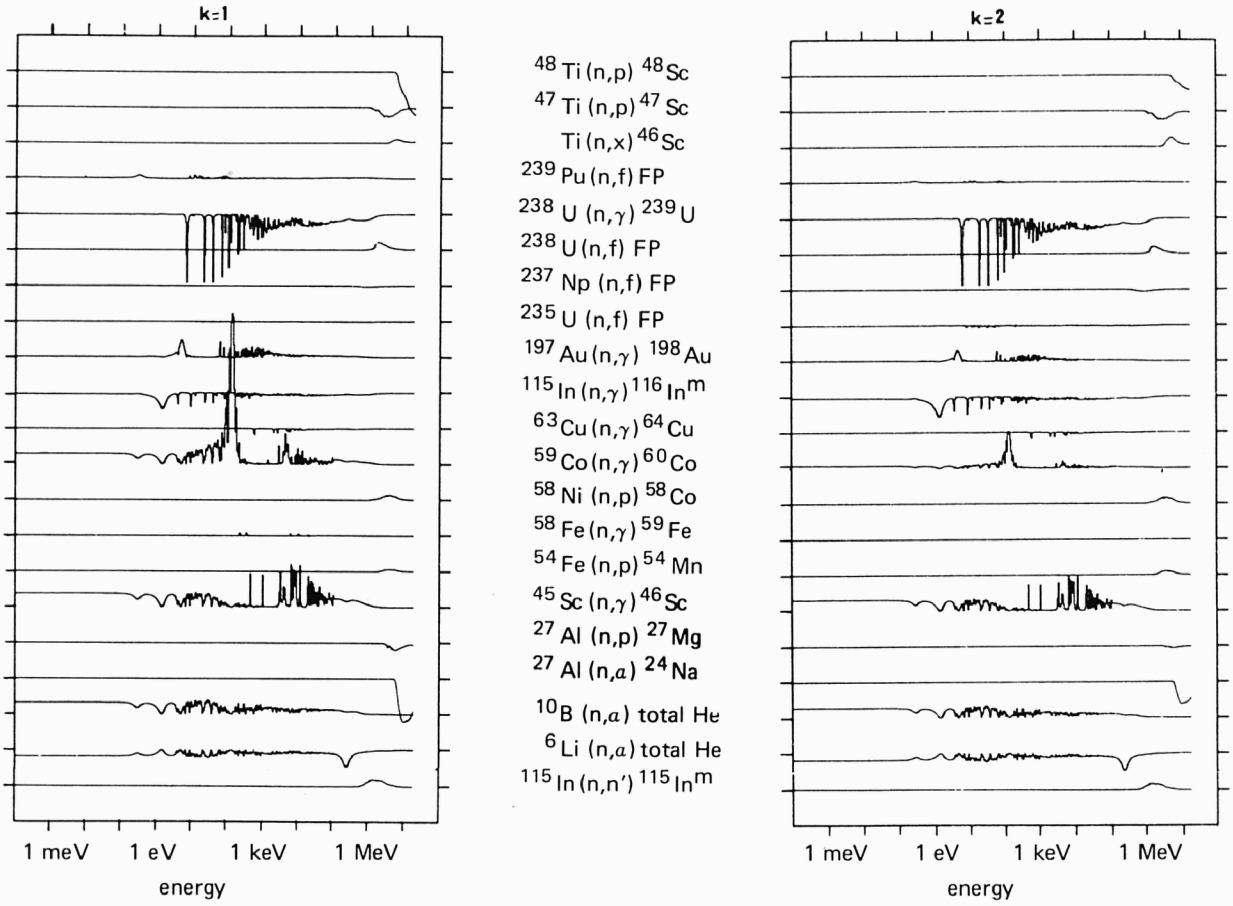


Fig. 6 Modifying factor for the CFRMF spectrum. The plots refer to the first and second iteration step.

1.2.5 Dismantling Cell

In 1977, the cell processed

- 48 specimen carriers from current irradiation programmes,
- 8 obsolete capsules,
- 84 HFR fuel element end pieces.

The cell ventilation radioactivity monitoring has been modernized, and studies commenced for a complete revision of the ventilation control system.

Work for a thorough overhauling of the dismantling cell was started in December, comprising

- decontamination and complete repainting,
- the replacement of the inner window,
- repair of the power manipulator,
- installation of additional lamps and tools. (Fig. 7).

1.2.6 Fuel Cycle

Uranium Supply

The US export license for 40,3 kg of ^{235}U was obtained in June 1977, in time to avoid reactor outages in 1978. A total amount of 39 kg ^{235}U has been ordered during the year.

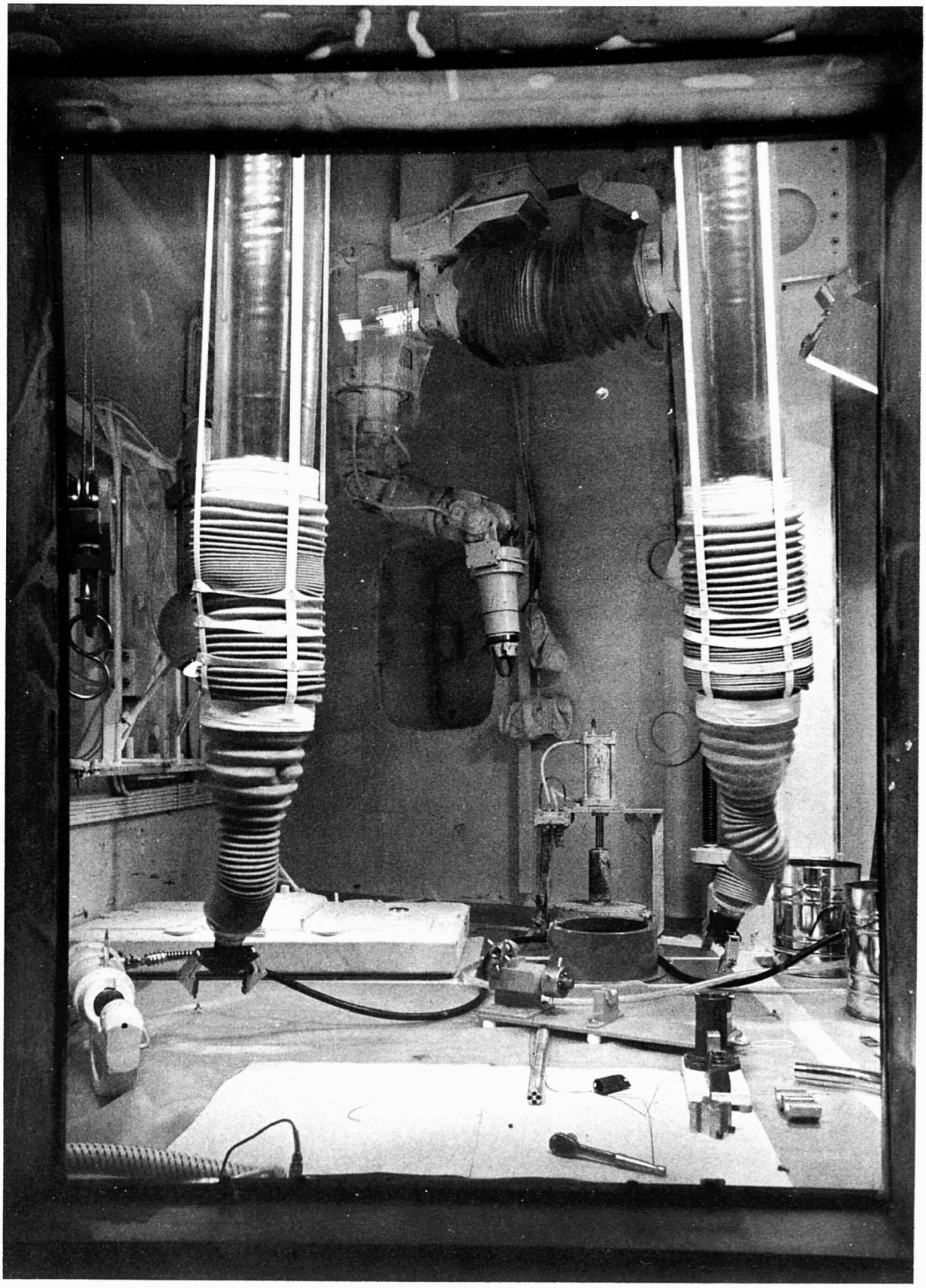


Fig. 7 *HFR Dismantling cell. Cell interior after the 1977/78 revision.*

Fuel Elements

A new fuel element manufacture contract has been signed in 1977, for four years of reactor operation. It includes a slight reduction of the burnable poison contents in the fuel element side plates (from 1,2 to 1,0 g ^{10}B).

The movement of fuel elements is represented in Table 4.

Studies have been carried out on the utilization of UAl-plate-type MTR fuel elements with 20°/o enriched uranium. Although reactor operation appears to be feasible the performance of HFR as an irradiation facility and its economics would largely suffer from the change-over.

Table 4 : HFR fuel elements. Movements to and from the Petten site.

	1976		1977	
	First half	Second half	First half	Second half
Transfer of depleted fuel elements	28	--	--	42
Transfer of depleted control rods	6	--	--	--
Average burn-up of transferred fuel	50°/o	--	--	49°/o
Average burn-up of transferred control rods	44°/o	--	--	--
Delivery of new fuel elements	55	24	55	37
Delivery of new control rods	--	17	--	18
New fuel elements available for use at end of reporting period	46	40	52	59
New control rods available for use at end of reporting period	8	16	9	20
New fuel elements charged to core	52	30	43	30
New control rods charged to core	9	9	7	7
Fuel element depleted	30	32	37	27
Average burn-up of depleted fuel	49°/o	48°/o	48°/o	51°/o
Control rods depleted	8	6	8	7
Average burn-up of depleted control rods	51°/o	47°/o	50°/o	49°/o

Reprocessing

A new contract covering all services of the external HFR fuel cycle has been signed in 1977. It implies among others, reprocessing of spent fuel elements in the Savannah River Plant, USA. The first transport of 42 elements left Petten in December 1977 (Fig. 8), and three more transports for 1978 have been prepared.

Fig. 8 *Transport of depleted HFR fuel elements to the Savannah River reprocessing plant. Shielded transport flask.*

2. UTILIZATION OF THE REACTOR

2.1 GENERAL

As in previous years the major part of the experiments carried out in and around HFR Petten was presented by materials testing irradiations for power reactor development programmes, followed by nuclear and solid state physics, and by radioisotope production. Table 5 gives a survey of work carried out during the year.

Table 5 : Status of experiments, and major progress achieved.

Project Nr.	Designation. Kind of experiment	Work carried out in 1977
ER 005	DRAGON graphite series	Post-irradiation examens of the last experiment
ER 006	Standard isotope facilities	Continuous utilization for radio-isotope production
R009,010,011, 013,014,107, 130, 159	Various nuclear physics and solid state physics experiments in the horizontal beam tubes	Continuous utilization
RX 043	CADO. Nuclear heating calori-meters	Regular measurements in various in-tank posi-tions
R 054	Fast reactor fuel over temperature tests (SHOT)	Rig. nr. 38 irradiated, nr. 37 started. Two new experiments under preparation (ECN)
R 063	Fast reactor fuel loss-of-cooling tests (LOC)	Rig. nr. 15, 17, 18 and 19 irradiated. Two new experiments under preparation (ECN)
E 084	Oxide fuel ultra-sonic thermometry (TRESON)	Design and manufacture of experiments nr. 3 and 4
D 085	Fundamental graphite properties investigation	Irradiation of rigs nr. 15, 16, 22, 27, start of nr. 17 and 23. Design and manufacture of three new experiments
ER 090	Reloadable isotope facility (RIF)	Continuous utilization for radioisotope production
RX 092	HFR vessel material neutron damage studies	Intermittent irradiation in various in-tank positions
E 094	In-pile high temperature sensor tests	Third irradiation started (interrupted due to difficulties with calibration thermocouple)
R 103	High pressure water irradiation facility (BERO)	Irradiation carried out
E 121	LWR fuel irradiation device development	Four short-term irradiations with special instrumentation

Table 5 (contd.)

Project Nr.	Designation. Kind of experiment	Work carried out in 1977
D 125	Power ramping of pre-irradiated LWR fuel pins	24 ramp tests completed. Various equipment manufactured
D 128	In-pile measurement of PCI on LWR fuel pins	First irradiation started
ER 136	Fissile target isotope facility (FIT)	Continuous utilization for radio-isotope production
R 137	Coated HTR fuel particle irradiation (BATAVIA)	Post-irradiation examens (PIE)
D 138	HTR spherical fuel element test	Start of the irradiation
R 139	TRIO steel specimen irradiations (SINAS)	Seven new irradiations. Following 10 rigs under manufacture
E 145	Fracture mechanics long-term steel irradiation (AUSTIN)	Irradiation continued throughout the year
R 151	V and Nb specimen irradiations (NIRVANA)	First experiments started. Interrupted due to unsatisfactory irradiation temperatures
E 154	Carbide fuel ultra-sonic thermometry (CARSON)	Design, manufacture, and irradiation carried out
D 156	Intermittent measurement graphite creep rigs (DISCREET)	Design, manufacture, and first irradiation
D 162	Coated HTR fuel particle amoeba effect experiment (ARTEMIS)	Design and calculation work
R 163, 164, 165	In-pile test facilities for the HFR TOP series	Design and calculation work (ECN)
D 166	In-pile graphite creep measurement rig (CRIMP)	Design and calculation work
E 167, 168	Steel specimen creep facilities	Design and calculation work
E 170	Carbide fuel pin profile gauge (POCY)	Design and calculation work
E 172	Oxide fuel corrosion studies (CORROX)	Design studies, specifications

The overall utilization of the reactor, however, lagged behind the scheduled occupation (59% vs. 70%) mainly due to

- work overload in the services directly concerned (project engineering, drawing and computing offices, workshops, electronics),
- delayed supplies,
- programme and/or design modifications on several experiments.

Table 6 compares the 1977 utilization to the figures of the last two years of the preceding JRC programme.

Table 6: Comparison of the 1975-1977 irradiation programmes.

Nature of the irradiation specimens	1975	1976	1977
Graphite, and HTR fuel	33	37	19
Structural Materials	13	16	25
LWR fuel	10	8	13
Fast breeder reactor fuel	10	8	5
Miscellaneous	19	10	8
Horizontal Beam Tubes, and Isotopes	15	21	30
Total, used capacity	100°/o	100°/o	100°/o
Reactor occupation in °/o of the theoretical maximum	52,5	67,9	59,3

2.2 IRRADIATION EXPERIMENTS

2.2.1 Graphite

With the post-irradiation examens of the last experiment, the longstanding ECN sponsored graphite series has been concluded in 1977.
The fundamental properties series for KFA Jülich continued with the following irradiations, (Table 7).

Table 7: 1977 Large Fundamental Graphite Irradiation Programme (D085).

Exp. Nr.	Irradiation Period	Irradiation Temperature (°C)	In-Pile Performance
15	December '76 - January '77	1250	Heat losses at the ends of the specimen carrier
16	September '76 - March '77	900 1050 1150	1150°C specimen carrier unloaded due to thermo-couple failures
17	November '77 - (March '78)	1100	Satisfactory
22	December '76 - December '77	300	Satisfactory
23	December '77 -	300 400 500	Temperature drift in the 500°C specimen carrier
27	March '77 - December '77	750 900 1050	Satisfactory

For studies of irradiation-induced creep in graphite, a new series (DISCREET) has been started in June 1977. It features cylindrical specimens kept under controlled tensile and compressive load (Fig. 9) at 300° and 500°C. The specimens are unloaded at intervals, and the creep strain is measured in a hot cell (Fig. 10) before being reassembled into a fresh irradiation capsule.

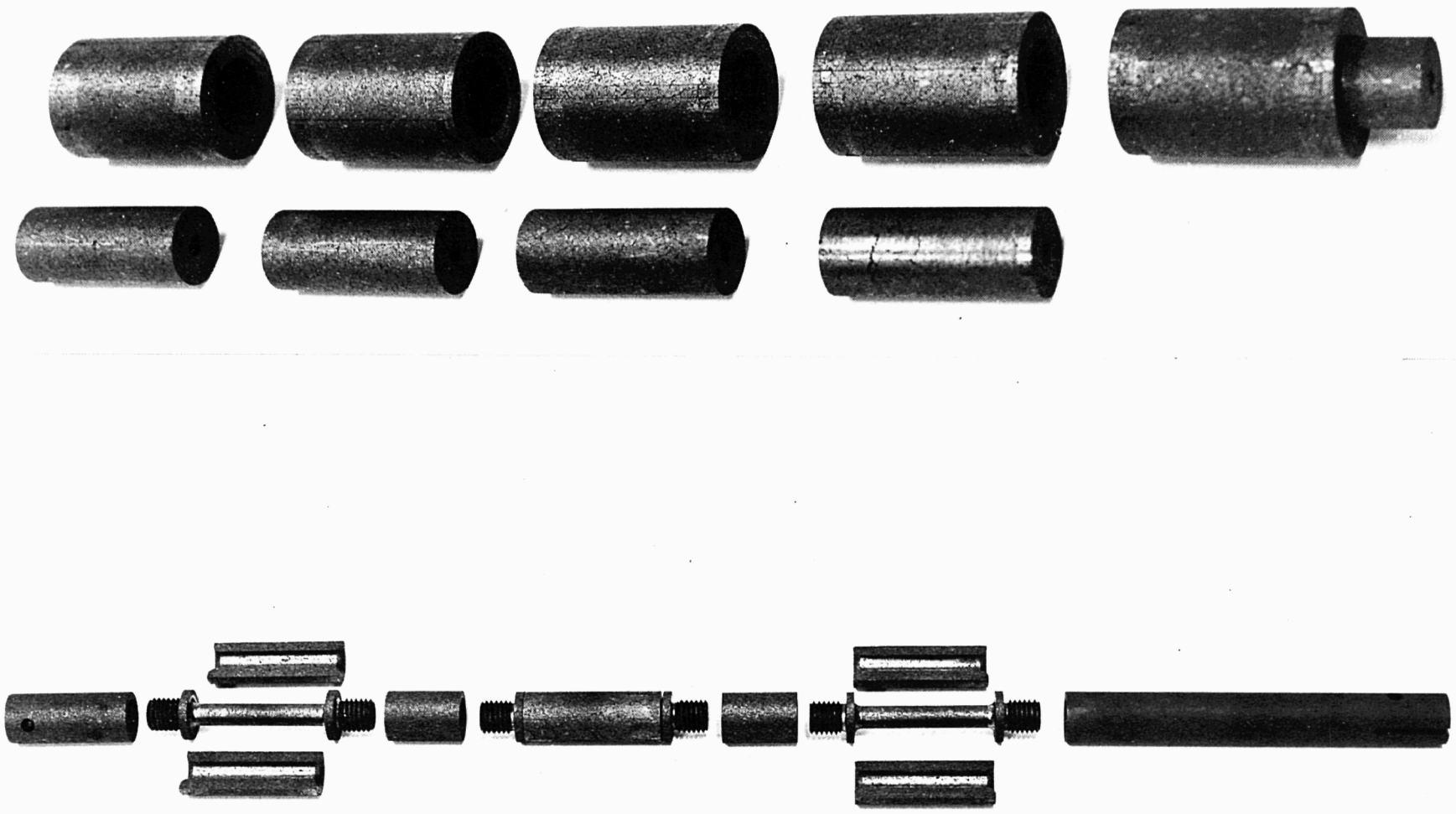


Fig. 9 *Graphite creep experiment "DISCREET". Compressive and tensile specimen stacks.*

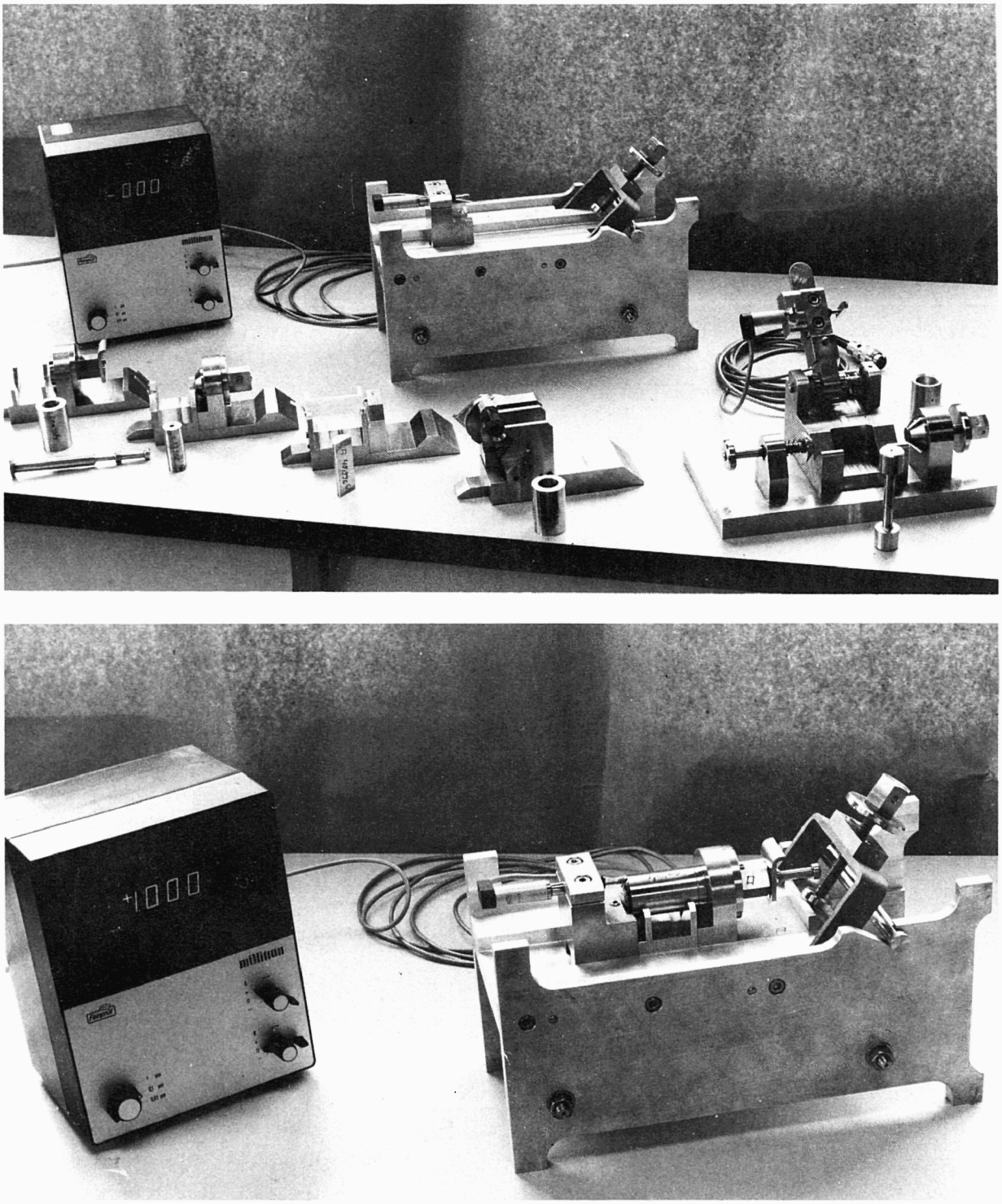


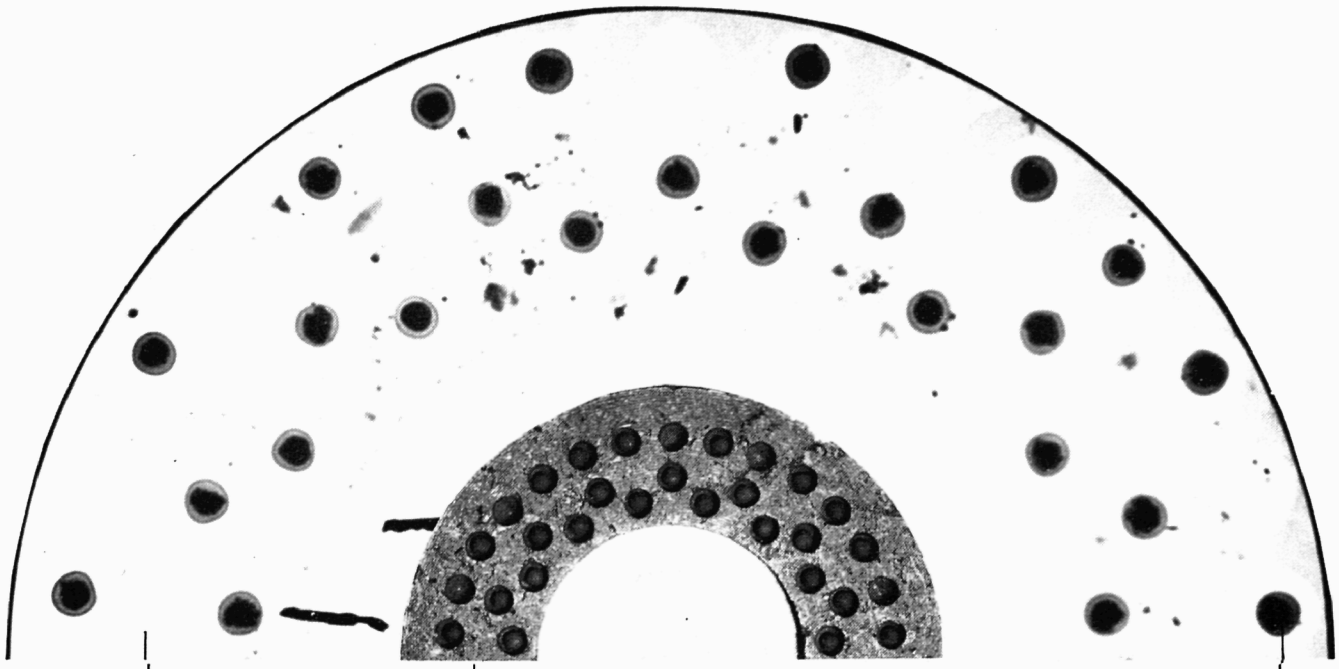
Fig. 10 Hot cell apparatus for the remote dimensional measurement of graphite samples.

2.2.2 HTR Fuel

The irradiation of various types of coated particles, at 750°, 1000°, and 1250°C (BATAVIA) has been terminated as scheduled. The data were

- irradiation time 286,4 days
- neutron fluence (> 0,1 MeV) $1{,}24 \times 10^{22} cm^{-2}$
- peak burn-up 73°/o fima

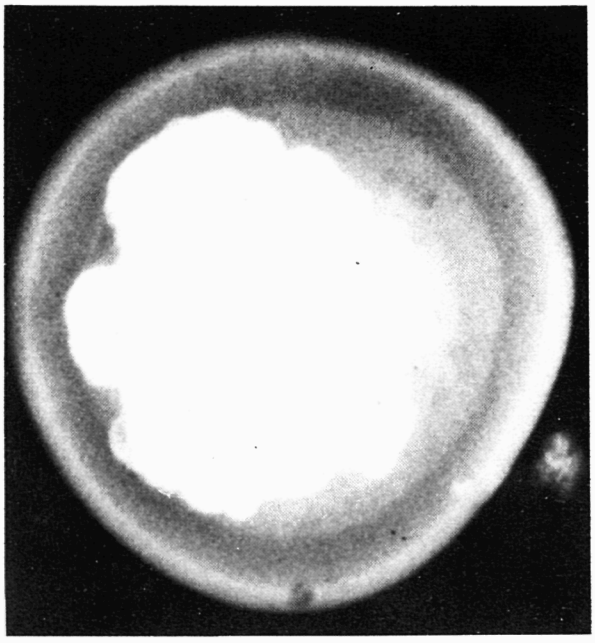
The non-destructive post-irradiation examens on the dismantled specimens were carried out. An example is given on Fig. 11.



visual examinations: no failure

microradiographical examinations:
kernel displacement in all particles

Fuel type	:	MA 1 $UO_2.Al_2O_3$
enrichment	:	92,6 °/o U-5
temperature	:	~ 1000 °C
fluence EDN	:	~ 5,9 x 10^{21} cm^{-2}
burn-up	:	~ 73 °/o fima



microscopical investigation of kernel radial displacement

range of observed radial displacement in this tray 12 - 41 μm.

Fig. 11 "BATAVIA" (R137). Appearance of fuel type MA 1 of tray L1-4-13.

A new capsule with four spherical fuel elements in two separately controlled carriers started irradiation in November 1977.
Subsequent HTR fuel experiments are under preparation.

2.2.3 Structural Materials

The ECN programme for the investigation of neutron-induced embrittlement of fast reactor vessel material continued in 1977 with the irradiation of seven TRIO capsules. New developments in this series have been:

- the increase of the number of specimens per TRIO from 5 to 15 which became possible after a slight reduction of the specimen head diameter
- the introduction of 4-cycle irradiations for GfK Karlsruhe ("K"-series)
- design and development work for block specimens (WOL and CT) for fracture mechanics studies. Fig. 12 shows the assembled specimen stack of the first CT capsule before loading into a REFA 170 standard capsule.

The irradiation of small tensile stainless steel specimens at 500°C in sodium for the JRC Ispra Reactor Safety Programme, continued throughout the year.
LWR fuel element grid and spacer specimens have been irradiation-tested in a pressurized water facility "BERO" (Fig. 13), at steady state conditions of 240°C and 80 bar. A neutron fluence of $3{,}8 \times 10^{21} cm^{-2}$ (> 1 MeV) has been accumulated during 9 reactor cycles.
A new irradiation capsule has been designed and developed for a large number of small niobium and vanadium samples. The specifications required irradiation temperatures of 450°, 600°, 725°, and 850°C, and an inert environment for the samples. Electron-beam welded Nb drums had been selected to house the specimens, protected by a thin-walled Nb tube. The irradiation of the first capsule which took place in August 1977, was plagued by uncertainties of the sample temperatures and by a sodium leak in one of the three specimen carriers. Post-mortem analyses by hot cell inspections and a number of computations revealed the necessity of major design modifications before a new irradiation can be started.
ECN are sponsoring a zircalloy creep-on experiment for the US NRC, Oak Ridge. The design and safety report has been issued in December 1977, the irradiation will start in 1978.

2.2.4 LWR Fuel

The large series of power variation experiments on pre-irradiated fuel pins in Boiling Water Fuel Capsules (BWFC) has been pursued in 1977: 24 ramp tests have been performed, together with extensive pre- and post-irradiation examens. First results of these tests have been published and discussed internationally. The second out-of-pile control equipment, installed in the reactor during 1977, now adds another four systems for BWFC experiments (Fig. 14). A specially instrumented device has been loaded in May 1977. It contains fission gas pressure, fuel pin length, and fuel stack displacement transducers connected to an LWR fuel pin.

2.2.5 Fast Reactor Fuel

ECN continued the experiments for research on SNR fuel pin safety, with the irradiation of two overtemperature capsules ("SHOT", see Fig. 15) and four loss-of-cooling devices ("LOC").

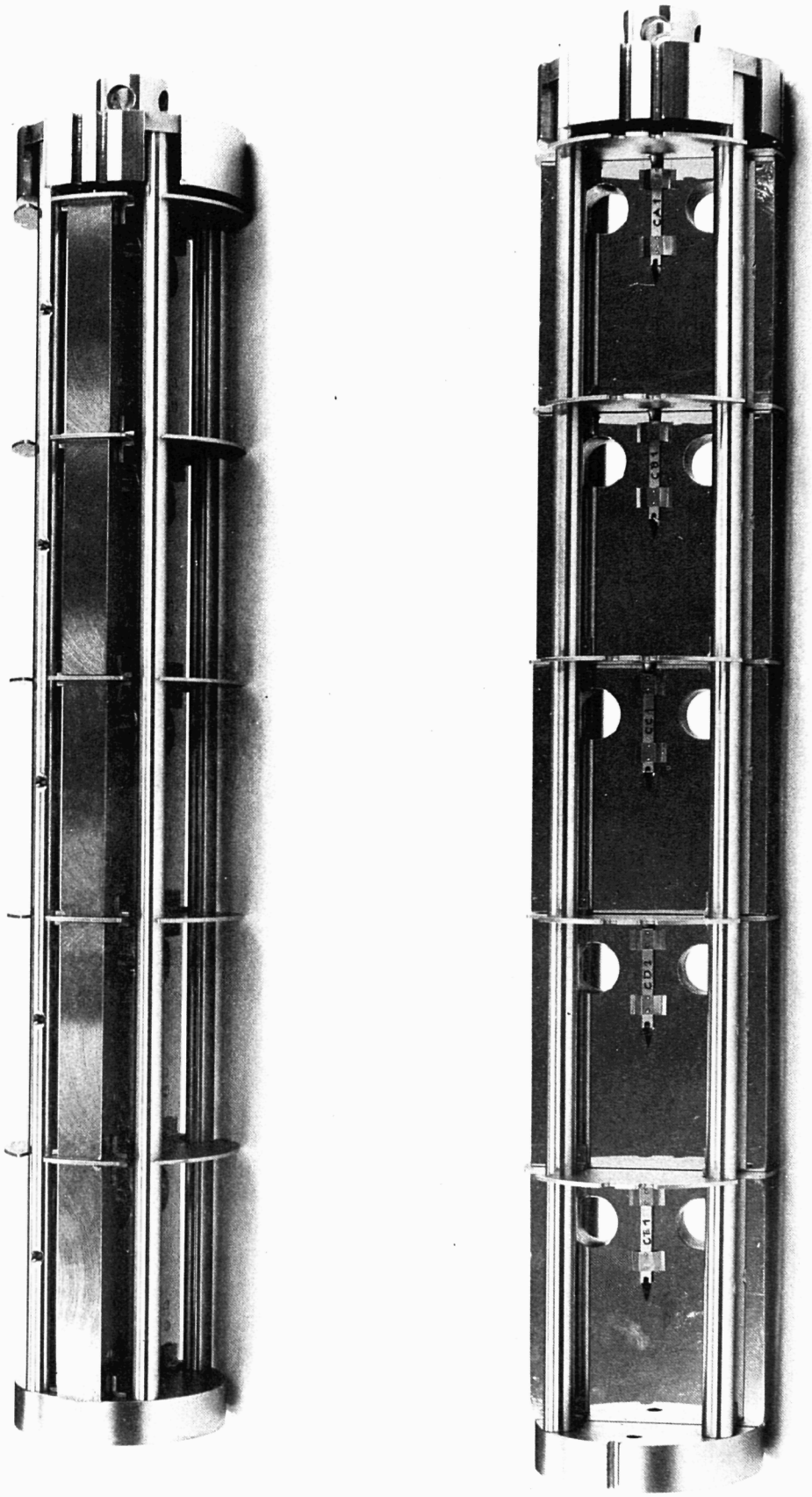


Fig. 12 CT specimen stack with base plates, baffles, tie rods and detector holders.

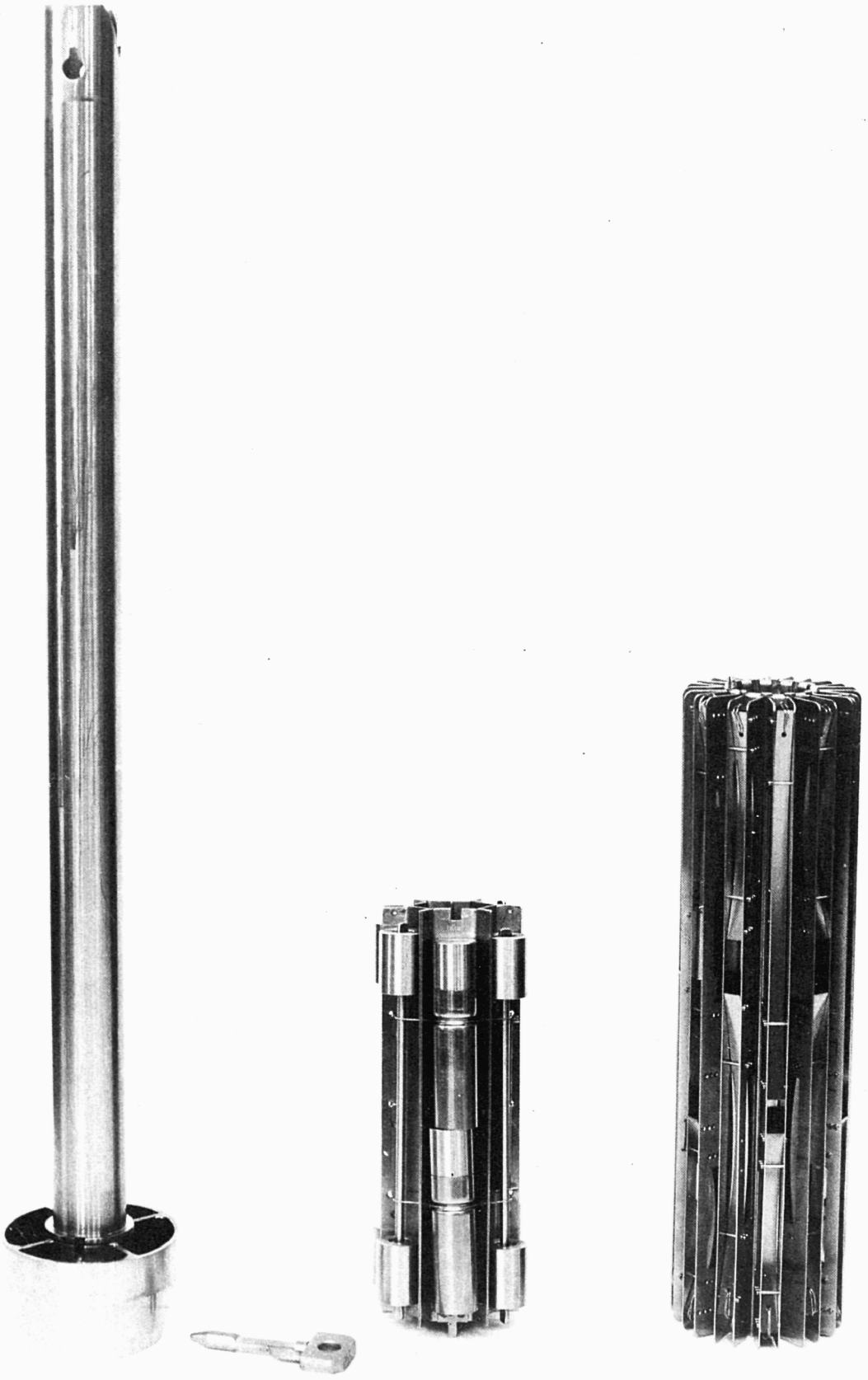


Fig. 13 Sample holder with samples irradiated in R 103 ("BERO", LWR fuel element components).

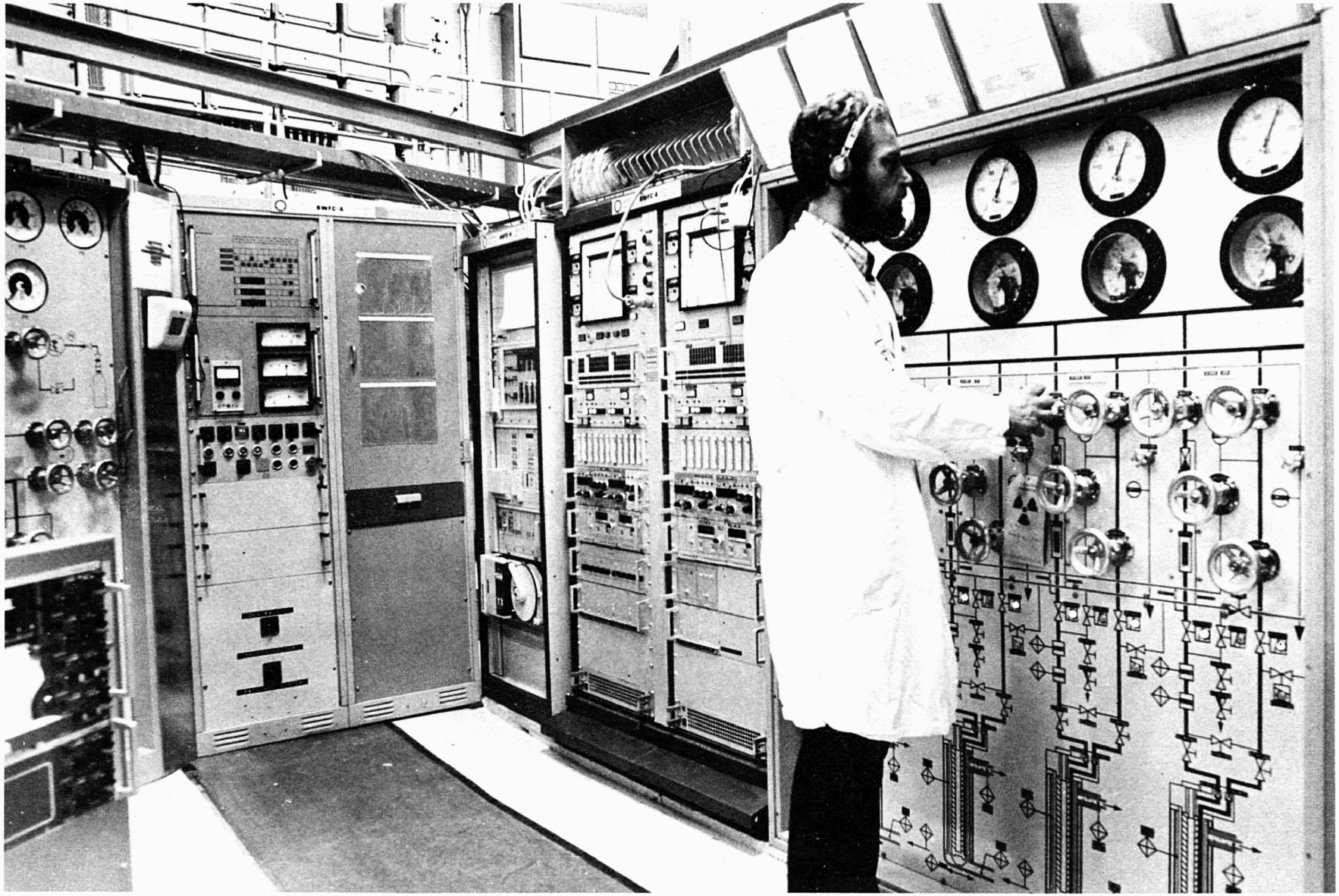


Fig. 14 BWFC out-of-pile installation.

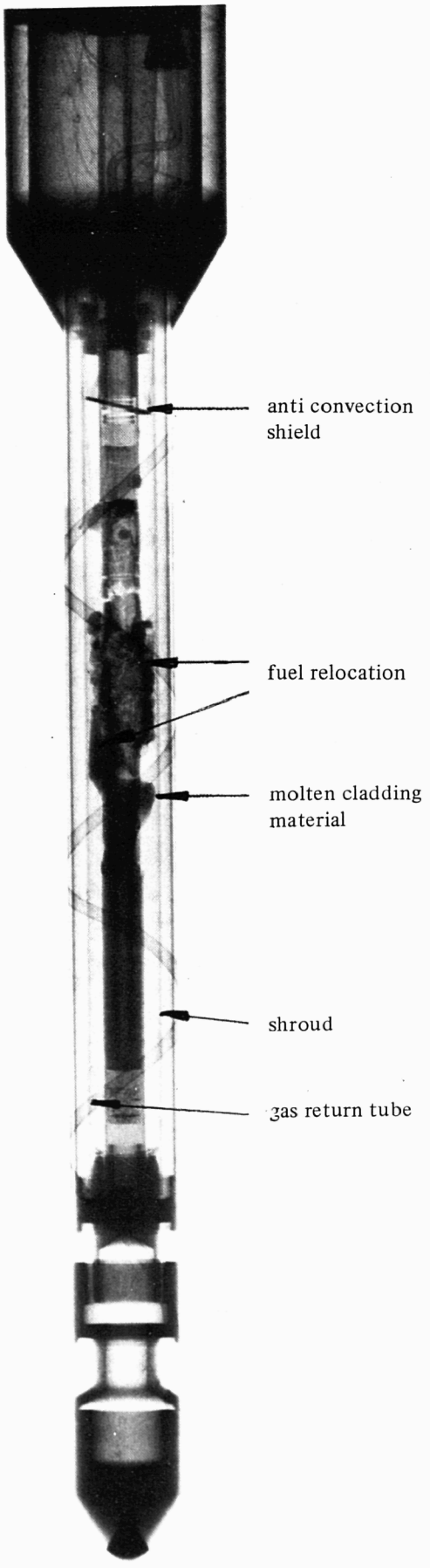


Fig. 15 Neutron-radiograph of SHOT capsule F38 after fuel pin failure.

Manufacture continued for two high power carbide pin irradiations, sponsored by GfK Karlsruhe, and new transient condition capsules were developed.
A special capsule with two ultra-sonic temperature sensors in mixed carbide fuel pins ("CARSON") has been manufactured and irradiated for JRC Karlsruhe.

2.2.6 Miscellaneous

Among various other projects the following should be mentioned:
- irradiation and hot cell examens of HFR vessel material (aluminium) surveillance specimens,
- regular nuclear heating measurements with the "CADO" calorimeter,
- three low-fluence irradiations of various plastic material specimens for JRC Ispra.

2.3 BEAM TUBE EXPERIMENTS

2.3.1 Solid State Physics

The operation of four horizontal beam tubes for solid state physics research was continued. Their employment for experimental investigations mostly carried out in close contact with university laboratories has been intensified.
At the single crystal neutron diffractometer at beam tube HB9 a helium cryostat has been installed by means of which crystals can be investigated at temperatures as low as 10 K.
In the experiments on the determination of spin densities in dilute alloys using the set-up for polarized neutrons also at beam tube HB9, procedures for crystal treatment and data analysis could be significantly improved such as to avoid or correct for extinction effects.
Investigations on solid state physics carried out by means of the HFR neutron beams were on:
- crystal and magnetic structures of inorganic substances (neutron diffraction),
- phase transitions in antiferromagnets, phase

diagrams, anisotropies and magnetic interactions in low-dimensional magnetic crystals (neutron diffraction and critical scattering),
- structure of liquid alloys (neutron diffraction),
- clustering in disordered alloys (diffuse scattering),
- polarization clouds in dilute alloys (diffuse scattering),
- spin glasses (diffuse scattering),
- spin densities in Ni-V alloys (polarized neutron scattering),
- structural phase transitions in incommensurate structures (neutron inelastic scattering),
- molecular rotations in solids (quasi-elastic scattering),
- spin waves around order-disorder phase transition in Fe_3Al (neutron inelastic scattering).

2.3.2 Nuclear Physics

Three horizontal beam tubes have been used to study neutron capture. All investigations were part of the research programme of Dutch universities and the investigators were organized in a joint group of ECN and the foundation FOM. This year the activities were concentrated on the study of nuclei in the sd shell (Na, Al and P) and in the fp shell (Ca, Sc, Ti, Mn, Fe and Ni) and on the fission product nucleus ^{141}Pr. Whenever feasible the target nuclei were polarized in order to study the spin admixture in the capturing state.

Fig. 16 shows the present nuclear orientation set-up, in which samples can be cooled down to 10^{-2} ^{o}K in a magnetic field up to 50 T. The purpose is to order the nuclear spins in the sample with respect to the direction of the spin of the neutrons. The gamma radiation which is emitted during the reaction is measured in several directions with the Ge(Li) spectrometers, which are also indicated in the figure.

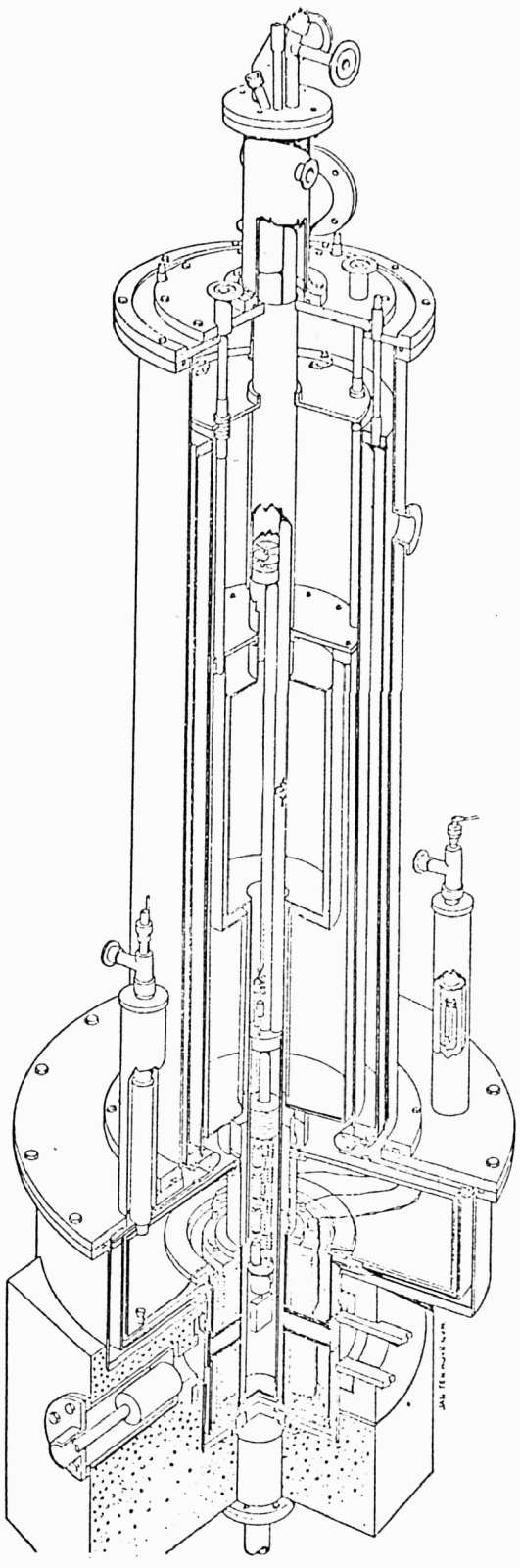


Fig. 16 Nuclear orientation experiment at 0,01^{o}K.

During the end of the year the beam tube HB0, which is situated at the large facility, has been taken into use. The set-up, which has been described in the 1976 Annual Report, has been improved with respect to shielding and has been equipped with better gamma spectrometers.

A very high flux density (7 x $10^8 cm^{-2,-1}$) can be reached at the focus point of this beam of pure cold neutrons, which is extracted by means of totally reflecting nickel mirrors. A special feature of the mirror system is that the mirrors can also be straightened to pass a very well focussed beam of unfiltered neutrons towards the focus point. In this beam several filters can be placed in a specially designed rotating drum shielding. In this way a neutron beam of energy equal to 24 keV is available for the study of resonances and of the neutron capture mechanism.

2.4 ISOTOPE PRODUCTION (Table 8)

As in previous years the production of ^{99}Mo constituted the major part of this activity. Both direct activation and production from ^{235}U have been pursued. The output from the activation production of ^{99}Mo could be increased by a higher loading of the capsules and by increasing the available neutron fluxes in positions D2/D8 and F2/F8. The fissile target ^{99}Mo production has been increased by nearly 50°/o as compared to 1976.
A number of electrical and electronic components for reactor control and safety systems have been exposed to ionising radiation in the Gamma Irradiation Facility.

Table 8 HFR radioisotope production. Occupation of the irradiation facilities.

Facility		Number of individual irradiations 1975	1976	1977
Poolside Isotope Facility	PIF	304	233	140
High Flux Poolside Isotope Facility	HFPIF	96	52	78
Reflector Isotope Plug	RIP	16	19	19
Reloadable Isotope Facility	RIF	562	678	633
Low Flux (rotating) Facility	PROF	173	190	175
Pneumatic Rabbit	PRS 1	1838	1549	1146
Hydraulic Rabbit	HR	14	19	9
Fissile Isotope Target	FIT	--	75	105
High Flux Isotope Rig	HIFI	--	15	--
Gamma Irradiation Facility	GIF	22	45	31
		3025	2875	2336

2.5 ACTIVATION ANALYSIS

For routine determination of trace elements in rock sediments and biological materials the rotating poolside facility (PROF) proved to be very suitable. The combination of neutron fluxes, irradiation times (up to 12 h) and cooling time (12 h) for handling is sufficient for the determination by instrumental analysis of about 10-20 elements in a sample. Before irradiation the samples are encapsuled in polythene vials of low impurity content. Each sample is accompanied by a small piece of iron wire to measure the neutron flux since these fluxes differ considerably depending on the place of the sample in the irradiation bottle. Concentrations of the trace elements are calculated by comparison with standards prepared at the laboratory. For control of the analytical results standard reference materials (NBS, IAEA)

are irradiated and measured in the same way.
Gamma spectra are recorded with Ge(Li) detectors and the output of the multichannel analyzers is processed by the CDC-6600 computer. The results from the computer are expressed in counts per μg for the element detected, and are corrected for radioactive decay and flux variations. Relations between samples based on trace element content can be established by another programme. This programme "CLUSTER" proved to be useful for the interpretation of geological and archaeological samples. Most of the samples are delivered by university geological institutes.
Trace element determination in water samples is of primary importance for environmental studies. A sampling and concentration method was developed based on collection of the trace elements on chemically activated charcoal. The charcoal treatment of the samples is carried out at the sampling site, so no losses or contamination occur before the neutron activation step. For low concentration of contaminants and high specific adsorption activated carbon was made from several pure organic materials e.g. sugar. Treatment of the organic polymeric material IXAN gave best results.
A study on trace elements in rainwater, specially in small fractions, was terminated with the defence of the doctoral thesis: "Neutron activation analysis of trace elements in rainwater" by J.B. Luten at the Utrecht State University on September 9, 1977 (Fig. 17).

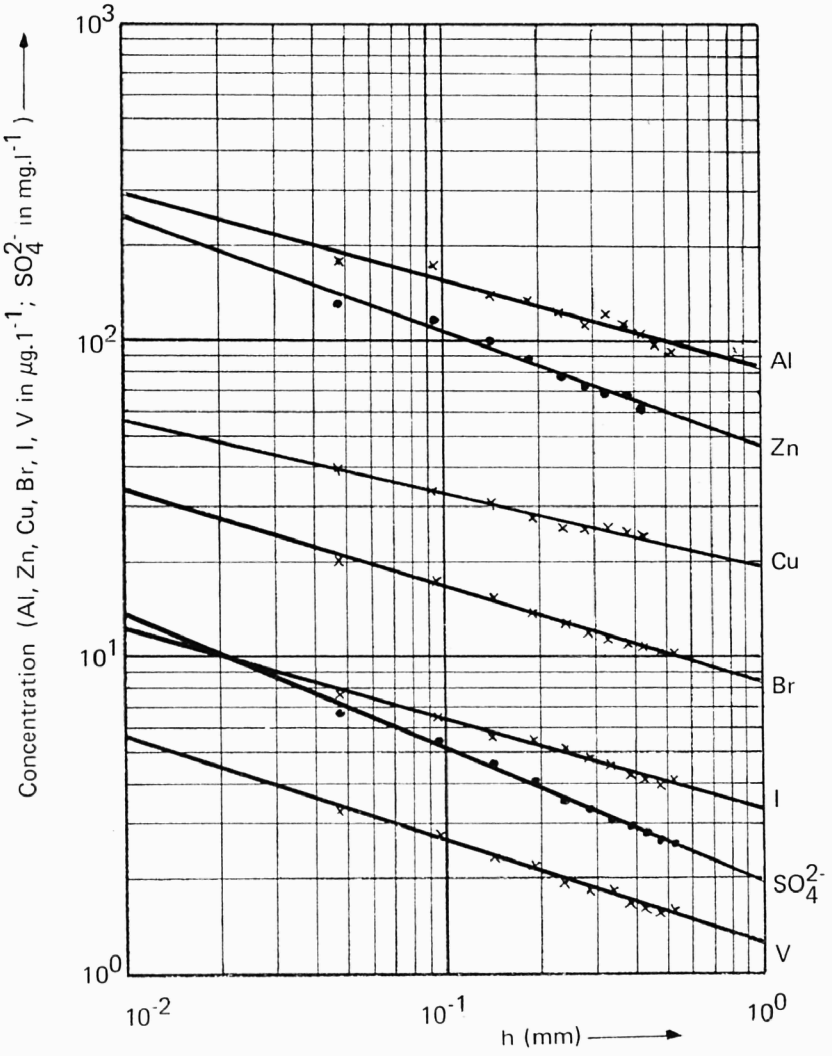


Fig. 17 *The concentration of Al, SO_4^{2-}, V, Cu, Zn, Br, and I during a rain shower, as a function of the depth of rainfall.*

II

MATERIALS RESEARCH DIVISION

- HIGH TEMPERATURE MATERIALS
- ORGANIC MATERIALS

INTRODUCTION

During 1977 the Materials Divisions activities in the field of high temperature materials have continued with emphasis towards establishing stable relations with other organisations on a collaborative basis, particularly in gas turbine, coal gasification and petrochemical areas. Notable has been the inclusion of certain activities in the COST 50 concerted action on materials for gas turbines together with membership of this group.

In the laboratories, attention has been focussed on equipment development for corrosion and mechanical properties and experimental work has been carried out in both these fields together with structural analysis in connection with these projects and under collaborative agreements.

The "HTM Advisory Committee for Programme Management" met for the first time on November 21st and 22nd at Petten.

Turning to the Organic Laboratories, work has continued as before with some extension of interests. An important achievement has been the certification of the first Petten sponsored reference materials and their storage on-site. These materials are intended for the calibration of flash-point apparatus in the petroleum industry. A laboratory has been equipped for work with potentially toxic materials and is at present used for work on polyaromatic hydrocarbon reference materials, work considered of Community importance by many national laboratories and organizations in Europe.

An important effort has also gone towards the technical and scientific support of other services of the Commission in the organic products sector.

HIGH TEMPERATURE MATERIALS

TABLE OF CONTENTS

Introduction

A new four years programme "High Temperature Materials"(HTM) has come into force for the Petten Establishment on January 1st, 1977. It is based upon the results of a Community-wide surveying "Review of Technological Requirements for High Temperature Materials R & D" which Petten had undertaken in 1975 and 1976 and which was concluded by the publication of a European "HTM White Book" under the same title (EUR 5623, September 1976).
The Petten HTM programme was confirmed by the Council of Ministers in its decision of July 1977 on the 1977-1978 Multiannual Programme of the Joint Research Centre*), as part of which it is intended to contribute to the Commission's future energy and industrial strategies by catalysing research and development in this field and by direct support of Community actions. Consequently therefore the HTM research activities constitute complements to Community national research, endowing the programme with the ability to provide a qualified centre for contact and co-ordination, the promotion of R & D in problem areas and the installation and operation of tests facilities.

Although originally subdivided into four fields of action according to the Council's definition, the programme has been reorganized in 1977 into the following five projects:

Project 1 - Meeting Point Petten
Project 2 - The effect of operational environment on mechanical properties
Project 3 - The relationship between structural and mechanical properties
Project 4 - The relationship between structural and physico-chemical properties
Project 5 - Major Test Facilities.

The project "Meeting Point Petten" has developed a central structure through which workshops, colloquia, training courses and international conferences are organized as "Petten Information Meetings" under participation of internal as well as external co-ordinators and scientific secretaries.

The programme's scientific activities are centred around the high temperature behaviour of materials under practical environmental conditions, so that creep and corrosion studies are conducted in typical atmospheres of the energy and chemical technology. This orientation also qualified the Petten Establishment for participation in the COST 50 concerted action**) to which the Council of Ministers has recently agreed.
36 research staff are engaged in this programme.

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*) *Official Journal Vol. 20, L/200/4 - 8.8.77 -*
**) *Co-operation in the domain of scientific and technical research. European concerted action - Materials for Gas Turbines COST 50 et seq.*

1. MEETING POINT

The Petten JRC Establishment has a remit to correlate high temperature materials research activities to be a coordinating element and a confluence of information on research carried out in European public institutions and industrial laboratories.
In order to optimize the communication between the Petten HTM programme and the current activities in research and development in industrial and technological practice, an information project called "Meeting Point Petten" has been established whose main objective is the provision of services in information to the high temperature materials community.

The spectrum of information needs of the European high-temperature community reaches from meetings devoted to oral exchange of information, surveying reports and further to data information systems. In order to structure the various activities in this project, the following 3 subprojectes have been defined to cover, respectively

- Conferences, Colloquia and Courses
- Surveys, Inquiries and Information Management
- Study on a High-Temperature Materials Data Bank

The success of the "Meeting Point Petten" activity, i.e. the creation of a place for concentration of public and industrial R & D, requires the knowledge of and contact with the existing European R & D potential, activities, programmes and institutions.
The following results complying with the defined objectives have been achieved in 1977, c.q.

- organisation of an international conference on Alloy 800,
- assessment of the Communitie's need for a high temperature materials data bank,
- organisation of training courses.
- due to continuous interest in the "High Temperature Materials White Book" a second edition of 400 copies was published in the course of 1977.

1.1 CONFERENCES, COLLOQUIA, COURSES

1.1.1 Alloy 800 Conference

The organisation of the International Conference on Alloy 800, which takes place on March 14th - 16th, 1978, has been set up. The conference will be concerned with the properties and the behaviour of Alloy 800 in energy systems and processes at elevated temperatures.
The session chairmen and review paper authors have been nominated and the preliminary programme prepared. The organisation of a post conference workshop on an Alloy 800 Data Bank has been started. The invitation and preliminary programme was printed and disseminated. This booklet is accompanied by a questionnaire on an Alloy 800 Data Bank. The answers will be evaluated and used as an input to the post conference workshop.
It is planned, that the conference programme committee formed by 14 invited experts from

the countries of the Community and staff from the General Directorate XII, XIII and JRC Petten, will meet for a final preparatory session at the end of January '78.

1.1.2 High Temperature Corrosion Course

Courses are initially designed for the training of Petten staff and will later be extended to include participation of others, with the final goal being to establish JRC Petten as a venue for HTM information meetings.
For 1978 the organisation of a series of courses for staff training is envisaged. The first course on "High-Temperature Corrosion" will be held from February 22nd to 24th. The preparation of this course is well advanced.

1.2 SURVEYS, INQUIRIES AND INFORMATION MANAGEMENT

The central aspect of this activity is the maintenance of communication with the European HTM community on specific aspects. One detailed objective is the revision at an appropriate time, of the "HTM White Book", either by supplements or by rewriting individual chapters. During the reporting period the official external publication of the "HTM White Book" took place (April 1st, 1977). A strong public interest lead to the publication of a second edition in the course of September 1977.

The results of a General Inquiry for Reference Materials, launched by the Community Bureau of Reference (BCR) in the member states have been compared with results obtained from the special HT-Reference Materials Inquiry presented in annex M of the HTM White Book. The need for HT Reference Materials in the member states of the Community are confirmed. The ACPM-BCR has therefore recommended on this basis the organization of a round table of experts to investigate the need for the organization of actions related to HT reference materials. This meeting will be organized by DG XII with the support of the Petten HTM programme.

An addresses file of organizations and firms within the HTM community has been established using magnetic tape to assist efficient handling, and is understood to serve as a first step in direction of the establishment of a "Central Information Index" which should in its final stage cover all current economic and research activities on high temperature materials in Europe.

1.3 STUDY ON A HIGH TEMPERATURE MATERIALS DATA BANK

In the course of the preceeding reporting period a study has been subcontracted to the Franklin Institute, Munich, with the following objective:

- provide and evaluate all basic information required to assess the need for a HTM data system in the Community,
- identify possible alternatives for future actions in the field.

In the frame of the study a combined interview/questionnaire contact was designed, containing the following elements:

- definition of materials/properties profiles based on an analysis of the types of data needed by industrial producers and users;
- identification and characterization of the users community as a function of their activities;
- identification of the present information sources and habits and location of institutions which maintain or plan to develop information systems for high temperature materials.

The results can be summarized as follows:

A. Characteristics of the HTM User community:

The representative HTM data and information system user is probably in an industrial or institutional research and development environment with a staff of 5 - 10 reporting to him. He probably has some management or executive responsibility. Primary amongst his professional responsibilities will be solution of problems surrounding the production of materials for, or their use in, gas turbines, chemical technology or new energy applications. Materials of major interest would be Ni-based alloys, austenitic and ferritic steels and high strength ceramics, in that order.

B. Present information sources and practice of the HTM community:

Typical of the information problems is inability to get up-to-date, evaluated data on a timely basis. In almost all cases data concerning service environment behaviour (application oriented information) and long time dependent test data are difficult to collect. The users rated corrosion in service environments, physical properties, strength, creep, applications data, research and development activity highest, with temperatures between 600^{o}C to 1000^{o}C and 1000^{o}C to 1500^{o}C of most importance and of equal ranking.
When asked to evaluate a HTM DB with respect to certain activities, new materials developments, reliability and failure analysis reflected most interested.

C. User reaction to the proposed HTM DB:

Of four possible information profiles given, the majority preferred those systems providing data as opposed to solely bibliographic information, e.g. abstracts. Off-line systems were generally preferred to on-line. Of HTM data users 3/4 were willing, under certain conditions, to contribute data and information to a HTM DB.

In conclusion of the results it seems reasonable to state that the HTM data users need application oriented, evaluated materials and property data. No single, generally available information system or service, meeting these needs, exists in Europe at this time and they would be pleased to see the development of a HTM DB.
It is estimated that within 400 institutions, around 800 - 1000 users could make use of an HTM-DB system.
The limited enthusiasm for an on-line service shown in the present study is probably due to the present situation. It does not preclude the use of such a system in the future. It is concluded that printed products (e.g. handbooks) should be the primary product in the early stages and that the HTM DB will be gradually shifted towards an on-line system.

For possible follow-up phases two tasks can be characterized:

- Resulting from the contacts made in connection with the conducted study, a momentum of interest has been created in the HTM user community and a feed back action in form of a report on the results of this study is planned.
- Development of a concept for the proposed HTM DB.

2. THE EFFECT OF OPERATIONAL ENVIRONMENT ON MECHANICAL PROPERTIES

2.1 THE INFLUENCE OF ENVIRONMENT ON THE CREEP PROPERTIES OF HIGH TEMPERATURE MATERIALS

Almost all designs of high temperature plant are based on the mechanical properties of the constructional materials which have been determined in air, but in many applications the high temperature alloys are exposed to corrosive gaseous environments, normally based on oxygen, carbon or sulphur. As a result the mechanical properties such as creep rupture life, creep strain rate, ductility etc., may be markedly influenced.
A particularly important class of environment is that containing carburising gases. Carburizing atmospheres can be encountered in almost all nuclear process and other chemical and power generation plant. High carbon potentials and temperatures up to about 1100°C lead to very strong carburisation of all heat resistant steels.
Considering the general situation two statements are important:

A. At temperatures of about 1000°C the creep behaviour, even under normal air conditions, is not well understood. The few experimental results existing are insufficient by far. Almost without exception, the data that exist relate to rupture life time.
B. The attack by carburising environments leads to very complex structural changes. Nothing about the type and the influence on creep of these changes is known at this time.

In 1977 experimental investigations were started that will subsequently enable the influence of a carburising atmosphere on the high temperature behaviour of heat resistant steels to be explored.
The tests which are being carried out in air will fill a gap in the general understanding of the creep properties at high temperatures for the materials concerned. Furthermore the data gained in air tests will be used as reference data for all other investigations to be performed in different corrosive environments.

By the end of 1977 the following creep testing equipment was available:

A. 8 single specimen creep machines are being used in air. All units have extensometers to allow continuous observation of creep strain.
B. There are 6 single specimen creep machines with similar equipment for the continuous measurement of extension, during testing in a corrosive gaseous environment. This type of machines is shown in Fig. 1.
C. 15 machines have been obtained which are capable of taking a ten specimen string enclosed in a working tube. These units are foreseen for life time measurements only.

The facilities mentioned under B) and C) have been put partially into operation in air. The experiments in gas will be started in the early part of 1978.

The experimental work has started in 1977 in air with a) the wrought 25Cr/Ni AISI Type 314 (German terminology 1.4841) and b) the centrifugally cast 25 Cr/20 Ni Alloy HK40.
The investigations concerning the 314 type steel have been carried out for a solution treated

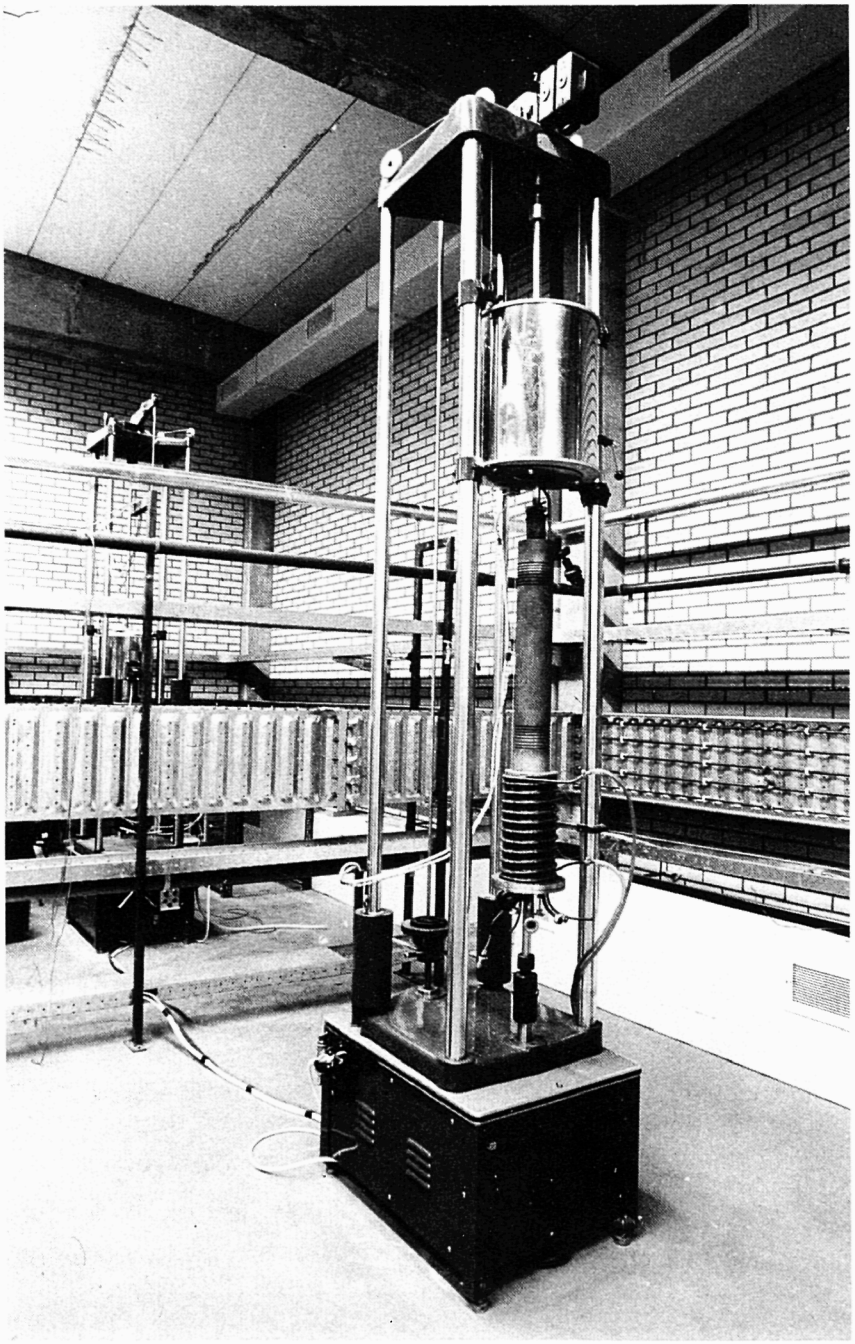


Fig. 1 Single specimen creep machine with gas tube.

condition and also on a 14%/o cold deformed condition. The deformed state has been incorporated because most steel used is subjected to some degree of cold deformation prior to service.

The experiments have been performed sofar at 900 and 950°C (on AISI Type 314) and at 900, 950, 1000 and 1050°C (on HK40). A test duration of about 200-300h has been chosen for most cases but some tests which would have a full lifetime of about 5000-10000h have been started. So far most experiments have been terminated when the minimum creep rate was reached and established and only a few allowed to run to the full stress rupture time.

A selection of creep tests results is given in Fig. 2, which shows the usual log minimum creep rate - log stress plots for the Type 314 and HK40 at 900°C.

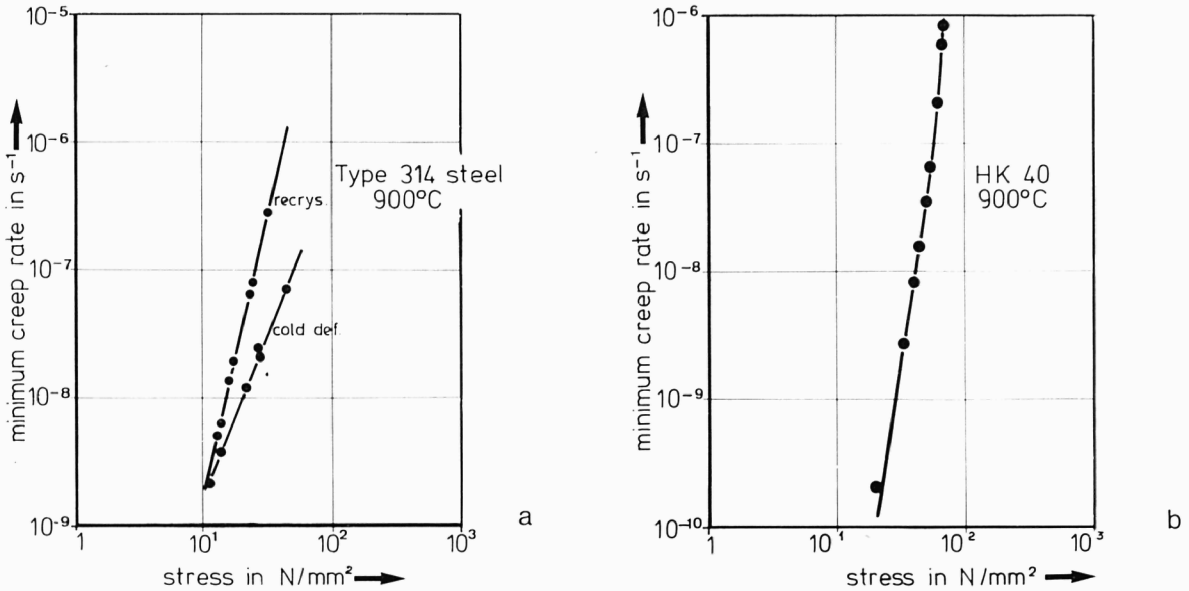


Fig. 2 Minimum creep rate vs. stress at 900°C a) Type 314 steel b) HK40.

For the Type 314 steel a linear dependence was found between the minimum creep rate and the stress over the whole stress range. The slope of the curve for the recrystallized material condition is about 4.5. The predeformed materials gives a slope of about 2.5. The results for HK40 however, show a discontinuity in the stress dependence. At low stresses, up to about 55 N/μm^2, the plot is linear with a slope of 6.4. At higher stresses the values again give a linear curve; however, the slope is about 12.3.

Structural analyses, especially by means of electron transmission microscopy, have been used to investigate the substructure of both Type 314 steel and HK40 after testing at different stresses. The development of precipitates during creep and the dislocation arrangement have been determined in detail in order to explain the mechanical test results and to get the basic information required to explain the further tests in gaseous environments.

Fig. 3 shows an example of the substructure of the Type 314 steel for a minimum creep rate of $10^{-7}s^{-1}$ reached after 178 h under a stress of 21,7 N/mm^2. A low angle grain boundary

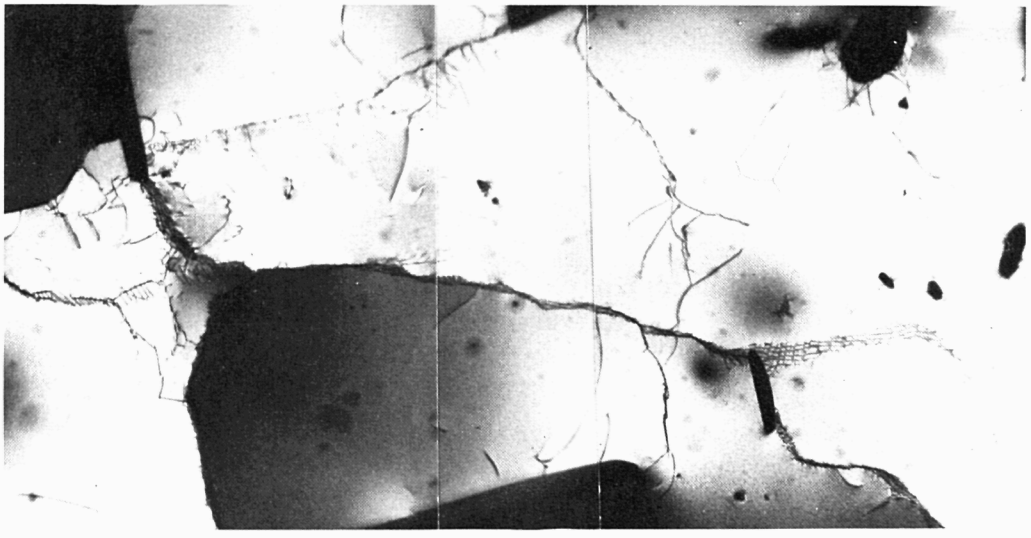


Fig. 3 Microstructure in Type 314 steel after reaching the minimum creep rate at 21,7 N/mm^2 at 900°C.

network has developed, which is partially pinned by carbides and σ-phases. The σ-phase seems to be very sensitive as a fracture source, as shown optically in Fig. 4 where the fracture path is

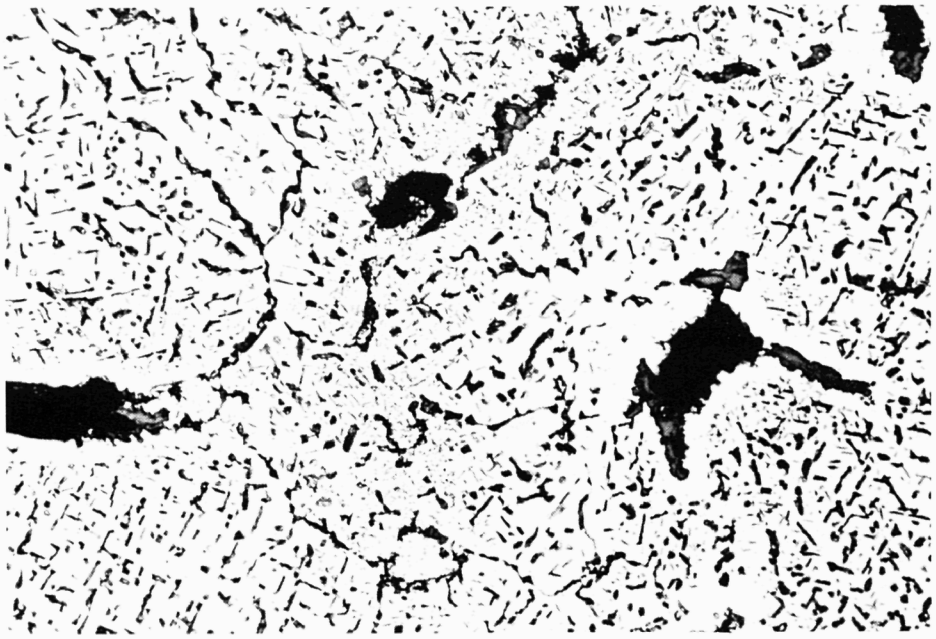


Fig. 4 Crack formation at σ-phase precipitates in Type 314 steel. Mag. 240

located in grain boundary regions where there are large σ-phase precipitates. Fig. 5 shows an example of the dislocation substructure of HK40 in the crept condition (stress σ = 44 N/mm^2, minimum creep rate $\dot{\epsilon}$ = 1,6 x $10^{-8}s^{-1}$). In comparison to the dislocation substructure of Type 314 steel (see Fig. 3), no subgrain boundaries have been developed. Only single dislocations are pinned by carbides.

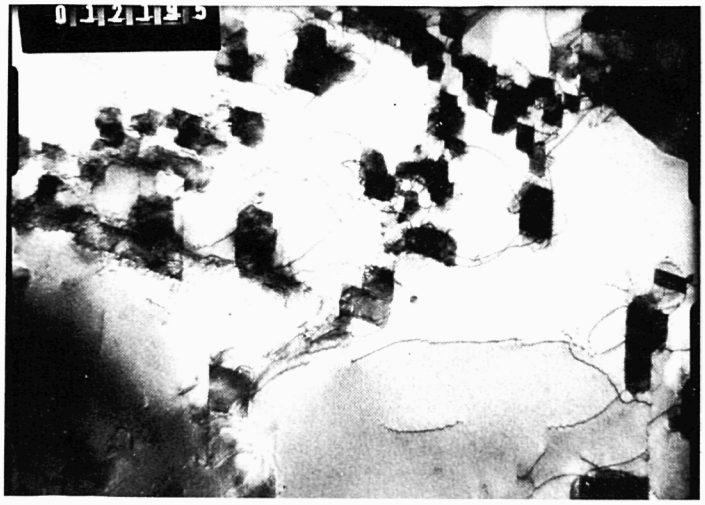


Fig. 5 Microstructure in HK40 after reaching the minimum creep at 44 N/mm^2 at 900^{o}C. Mag. 40.000.

2.2 A STUDY ON THE EFFECT OF COATINGS ON THE HIGH TEMPERATURE MECHANICAL PROPERTIES OF SOME NICKEL-BASE ALLOYS

Evidence is now available that the benefits derived from the improved corrosion resistance provided by a coating may be offset to some extent by a reduction in creep strength and fatigue properties of the coated component, so that the maximum extension of component life is not obtained. The scope of this work is to clarify the underlying damage mechanism associated with coating gas turbine blades and to evaluate factors presently ignored in the materials specification and fabrication codes.

The alloy IN100, a commercial low Cr cast Ni-base alloy widely used for aircraft turbine blading, has been selected for this study. The heat treatments applied to the as-cast alloy are

designed to simulate deposition of complex Cr-Al-coatings from the vapour phase.

Examination of microstructural features is carried out by means of optical microscopy, SEM and TEM, X-ray diffraction and AUGER/ESCA, and in addition physical property determinations are used whereever relevant to explore the kinetics of the process involved.

Research effort during the period under review was concentrated on:

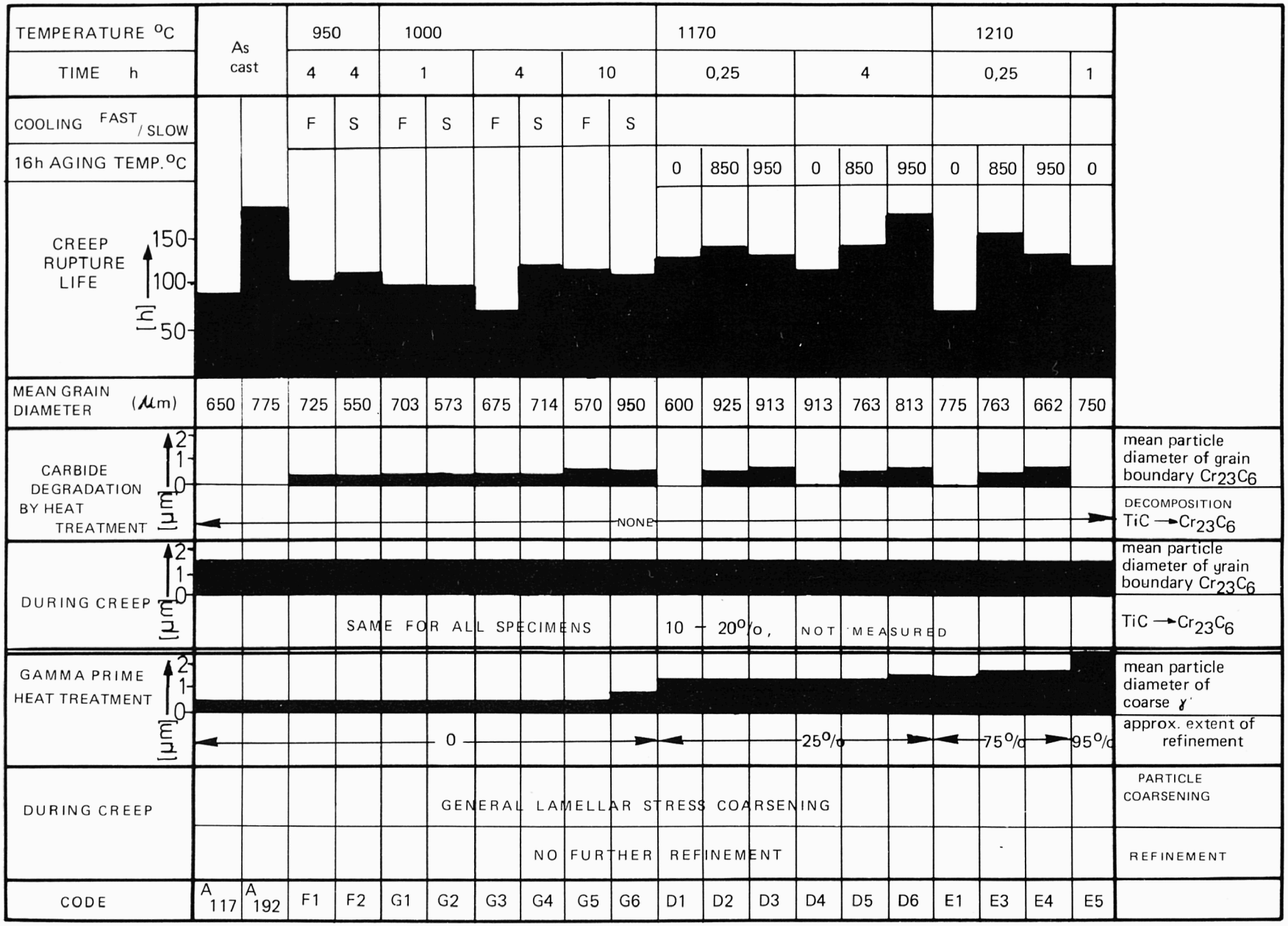
i) the evaluation of alternation of microstructure and physical properties induced by a variety of heat treatments which were designed to simulate different coating procedures;
ii) the microstructural investigation of a series of simulatively heat treated specimens which had been creep-tested in air.

The results of the microstructural investigation of a set of heat treated specimens which had been creep tested in air at 950°C under uniaxial load of 220 N/mm^2 are summarised in Table 1, together with the respective creep rupture lives. A preliminary examination of these results reveals that during creep exposure coarsening of all carbides has taken place together with a partical dissolutioning of the MC-type carbide (TiC) and concommitant precipitation of more $Cr_{23}C_6$ which migrates to grain boundaries in a film like morphology in addition to precipitating close to the original MC carbides (Fig. 6).

Fig. 6 Coarsening of MC-type carbide and appearance of small $M_{23}C_6$ precipitates during creep testing. (950°C/ 220 N/mm^2.

Further, the small cuboid gamma prime coarsens appreciably into lamellae (Fig. 7) in the highly stressed area of the specimens tested at 950°C. Grain structure and porosity appear to remain unaffected by the creep testing. Within the set of heat treated and creep tested specimens investigated, there appears to be no relationship between the given creep properties and the observed variations in microstructure and physical properties. Initial studies of the fracture behaviour suggest that the local grain size and the distribution of microporosity has the greatest influence on rupture life. These parameters have not yet been quantitatively assessed.

Table 1 : Variation of microstructural features of IN 100 by heat treatments and creep testing.



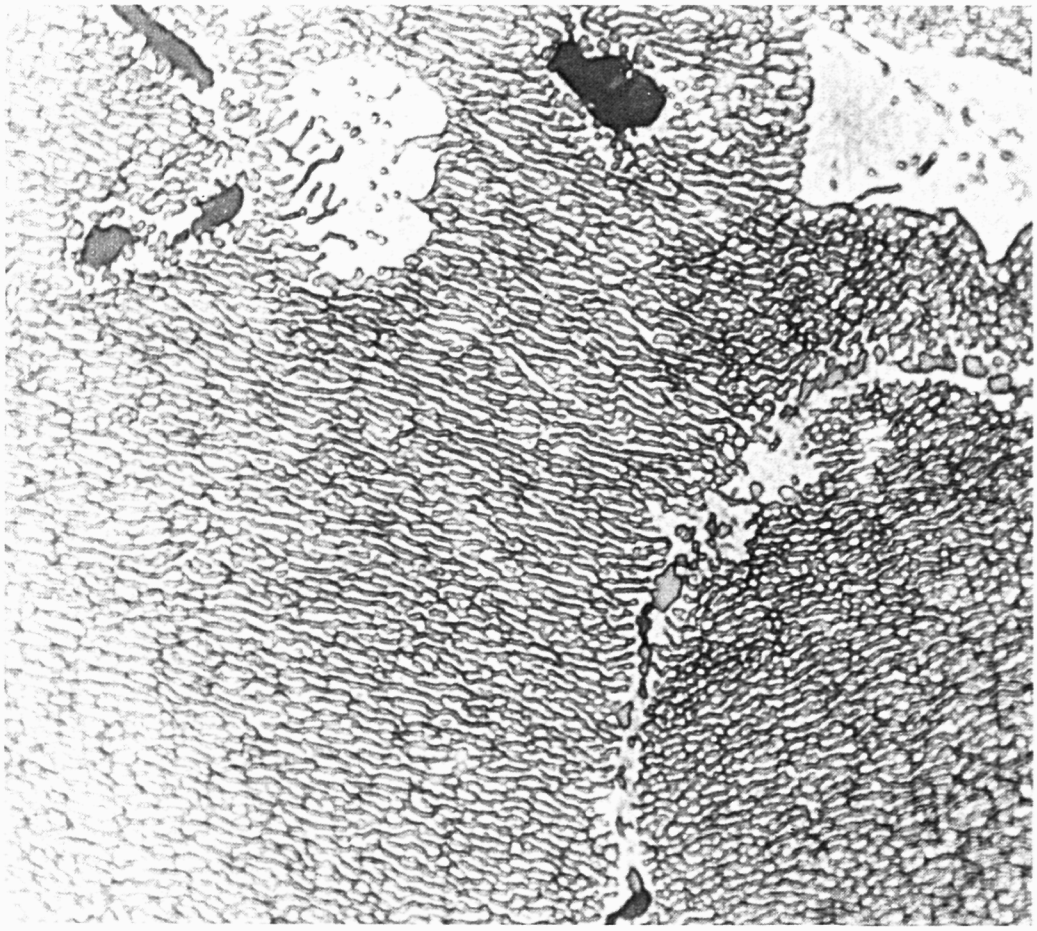


Fig. 7 Coarsening of cuboid gamma prime into lamellae during creep exposure. (950°C/ 220 N/mm^2).

In addition to the structural investigations on creep tested alloys, some kinetic studies of the precipitation process under long time ageing at high temperatures were performed by means of electrical resistivity measurements. It was concluded that in a solution treated (1120°C) and quenched specimen the equilibrium solid solution concentration of carbon is achieved during ageing at 850°C for one hour. This is followed by a long term resistivity decrease which can be related to the transformation of the MC-type carbide into $M_{23}C_6$. The resistivity results that are obtained when ageing is continued at temperatures in the range 950°C to 850°C suggest that the interactions between MC, $M_{23}C_6$, γ and γ' are both complex and sluggish.

The modification of the creep controlling structural features resulting from the various heat treatments and from the high temperature exposure during creep testing follows the same pattern as has been observed by several authors working with this or similar alloys. The corresponding creep behaviour, however, shows no indication of systematic variation with these changes in microstructure in the studies carried out so far.

3. THE RELATIONSHIP BETWEEN STRUCTURAL AND MECHANICAL PROPERTIES

The primary objective of the work in this project is to identify and determine the relative significance of the various factors which contribute to scatter in the measured creep and stress rupture properties of typical high temperature alloys; so meeting one of the requirements for the analysis of material performance in service.

The factors contributing to scatter may be divided into "intrinsic" and "extrinsic". "Intrinsic" factors refers to test sample material variations, whereas "extrinsic" factors are associated with variations in testing parameters such as temperature oscillations and gradients, stress variations, change in axiality of loading, shock loading of samples, etc.

A. "Extrinsic" factors affecting creep results

The effects of changing test procedure parameters on creep results have been analysed and the analysis has served as a basis for the selection of high precision testing equipment and measurement techniques.

In a typical superalloy, a load accuracy of $\pm$ 1 $^{o}/o$ (a current value for creep testing machines conforming to DIN standards) and a sample diameter tolerance of 0,05 mm (normal machine shop tolerance) can result in scatter in creep rate amounting to 34 $^{o}/o$. Even when sample diameters are accurate to $\pm$ 0.005 mm and load to $\pm$ 0.5 $^{o}/o$, creep rate scatter can still amount to 11 $^{o}/o$. The temperature influence on the creep-rate or rupture time scatter is shown in Fig. 8 for a typical superalloy.

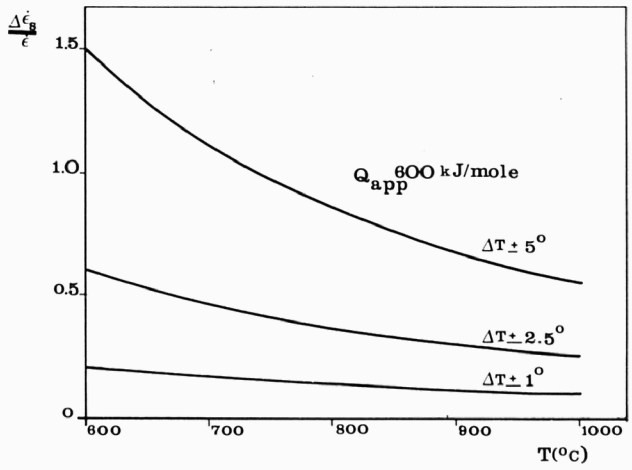


Fig. 8 *Influence of temperature inaccuracy on scatter in steady state creep rate.*

Comparisons of temperature measurement technique between different laboratories specialized in creep testing have revealed that actual deviations of 10°C from a common standard are not uncommon.

Temperature oscillations in time of about $\pm$ 2.5°C can reasonably be expected in single machine tests unless stringent precautions are taken with respect to the temperature control and measurement procedures followed. It is therefore to be expected that the scatter commonly observed on creep data is at least partly caused by temperature effects.

A third main effect which can strongly contribute to scatter in creep data is associated with the superposition on the uniaxial stresses of bending stresses resulting from excentric loading. Experimental data as to the extent of this effect have not been found in literature for superalloys. Model calculations in literature however, show that the effect on scatter can be substantial. For a constant eccentricity of $\delta/d = 0.01$ (corresponding to normal machine shop tolerances on a 6 mm diameter sample) and a stress exponent $n \cong 8$, the rupture time is

reduced to approximately 50°/o of the purely uniaxial value. In order to substantially reduce scatter as a result of eccentric loading the problem of misalignment has therefore to be tackled at its roots, i.e. in the testing machine itself as delivered by the manufacturer.
The first testing machine, designed for this type of high accuracy work, has been commissioned. A special loading technique to minimise bending stresses in the test piece is now being developed.

B. **Materials variables affecting creep results**

A detailed microstructural study has been made on a set of Waspaloy samples subjected to quality acceptance creep tests in industry and exhibiting a wide scatter in creep properties. Maxima to minima mean creep rate ratios of 9 and 13.7 have been observed for the most widely scattered single heat and between different heats respectively.

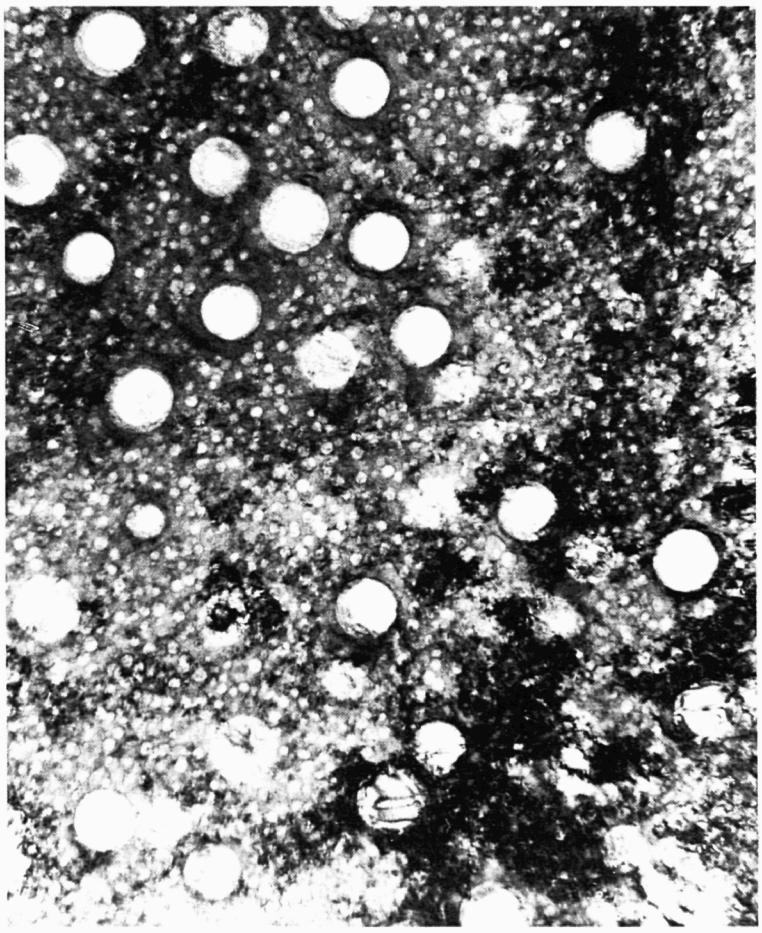
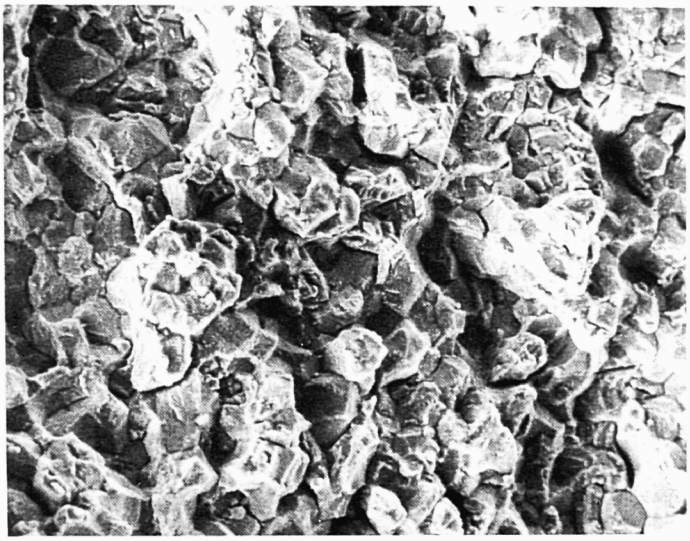


Fig. 9 *TEM micrograph of a crept Waspaloy sample showing a two size distribution of γ' particles. Magn. x30.000.*

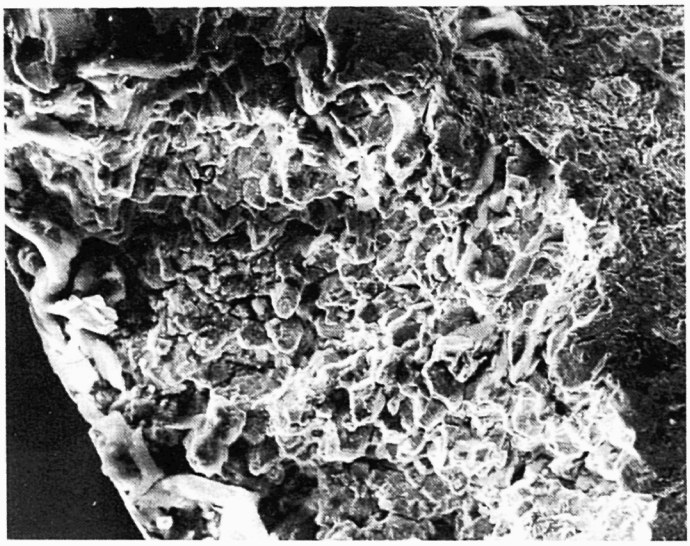
Waspaloy is strengthened primarily by the ordered fcc γ' precipitates and Fig. 9 shows it to consist of a two size distribution. The largest particles have a diameter of approximately 2500 Å in size, the smaller ones are nearly 500 Å in size. The same distribution, with similar dimensions, is observed in all samples (cf. Fig. 25).
Grain boundaries are strengthened by a discontinuous $M_{23}C_6$ carbide precipitate. Although TEM work did not reveal other grain boundary phases, X-ray analysis of extracts showed small quantities of Ti nitrides to be present.
No qualitative variations in microstructure were observed by TEM which could explain the large differences observed in creep rupture times. Optical metallography, however, revealed distinct differences in grain size. These were confirmed by SEM observations. Some definite differences in fracture mode were observed between samples with short rupture times ($\approx$ 50h) and samples with longer rupture times.

In the first case, the fracture surface showed a definite inter-granular appearance over a large part of the cross section (Fig. 10a). The exposed grain boundaries did not show any sign of plastic deformation. This typical decohesive rupture appears to have started at the surface of the sample and progressed inwards (Fig. 10b).
The remainder of the fracture surface shows evidence of a plastic, dimpled rupture with rather shallow dimples. Fig. 10c shows a transition area between the two modes of fracture. Specimens with longer lifetimes did not show intercrystalline fracture over large areas. Rather their fracture surfaces show a mixed mode of rupture with some crystalline facets visible in some areas but also signs of dimple rupture all over the cross section.

Some additional SEM and TEM work is still necessary in order to establish structural differences, if any, at the grain boundaries. Also the possibility of grain boundary segregation of foreign elements, which promote intergranular oxidation, can not be ruled out as a cause of the wide ranging time to rupture in some samples.



a



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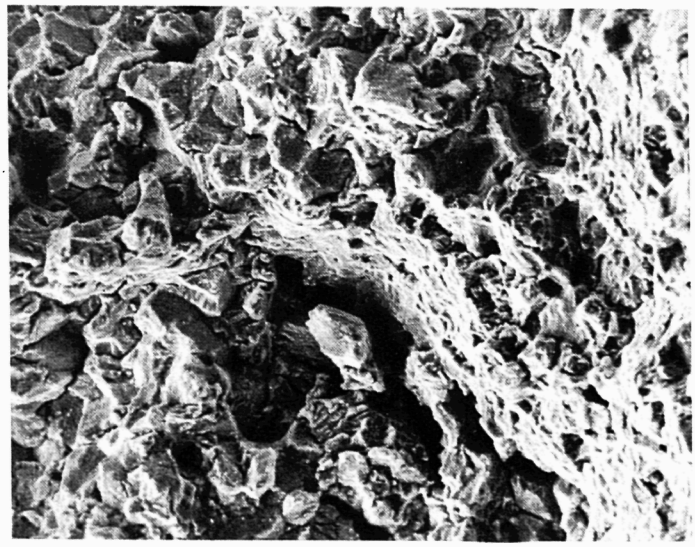


c

Fig. 10 SEM micrographs of the fracture surface of stress ruptured Waspaloy samples.

4. THE RELATIONSHIP BETWEEN STRUCTURAL AND PHYSICO-CHEMICAL PROPERTIES

4.1 CORROSION BY GASEOUS ENVIRONMENTS

Acceptance and effective utilisation of materials for the many developing petrochemical and coal conversion processes has been limited by a lack of detailed understanding of the principles by which alloy composition can influence corrosive attack in the complex environments involved. The objectives of this research programme are therefore directed towards understanding the various parameters which affect corrosion behaviour both in the basic carburising and sulphidising environments and also in the more complex gases found in some of these industrial processes. In particular the relative importance of alloying elements in promoting good corrosion resistance under these conditions must be established and linked to the industrial situation by the post-exposural examination of corroded materials extracted from commercial plant. The production of experimental data for these studies started during the period of this report.

A. Thermodynamic data

In order to interprete test results obtained from laboratory corrosion rigs, it is necessary to have an understanding of basic gas phase thermodynamic equilibria and metal stability diagrams derived initially from theoretical calculations. This preparative stage has been largely completed, Figure 11 serving as an example of the type of metal stability diagram constructed

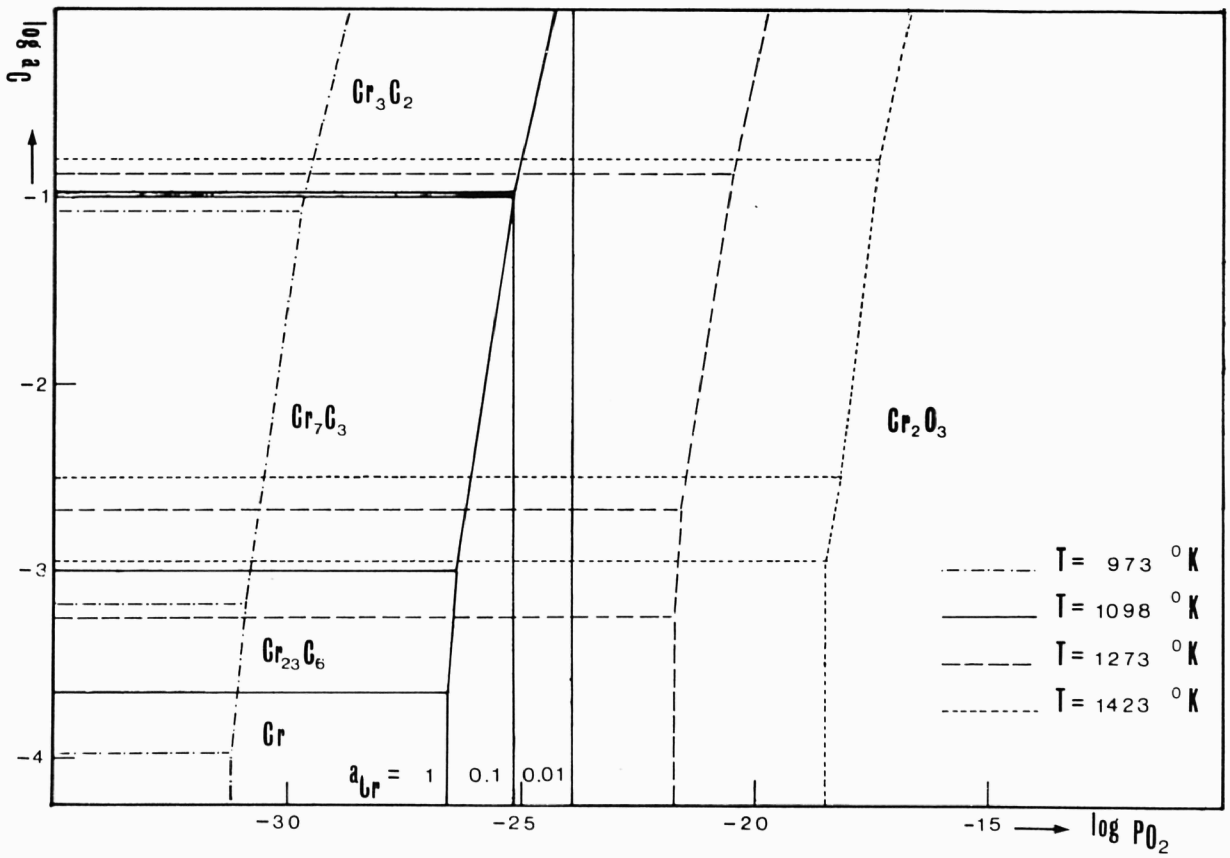


Fig. 11 Metal stability diagram for chromium in oxidising/carburising environments.

from theoretical considerations. It can be seen that a range of relevant metal activities and temperatures have been considered.

B. Carburisation/Oxidation

During corrosion testing the kinetics of the various corrosion processes are being monitored, in the first instance, by gravimetric techniques, i.e. determining weight changes due to the ingress of carbon, oxygen or sulphur from the gaseous environments. These changes will be monitored either discontinuously, necessitating the periodic interruption of the tests, or continuously, in-situ, using a high sensitivity thermobalance. Several designs of corrosion test autoclave have been devised to meet these various requirements and also the additional one, that of reliably containing the hazardous, corrosive test environments at elevated temperature for long periods of time.

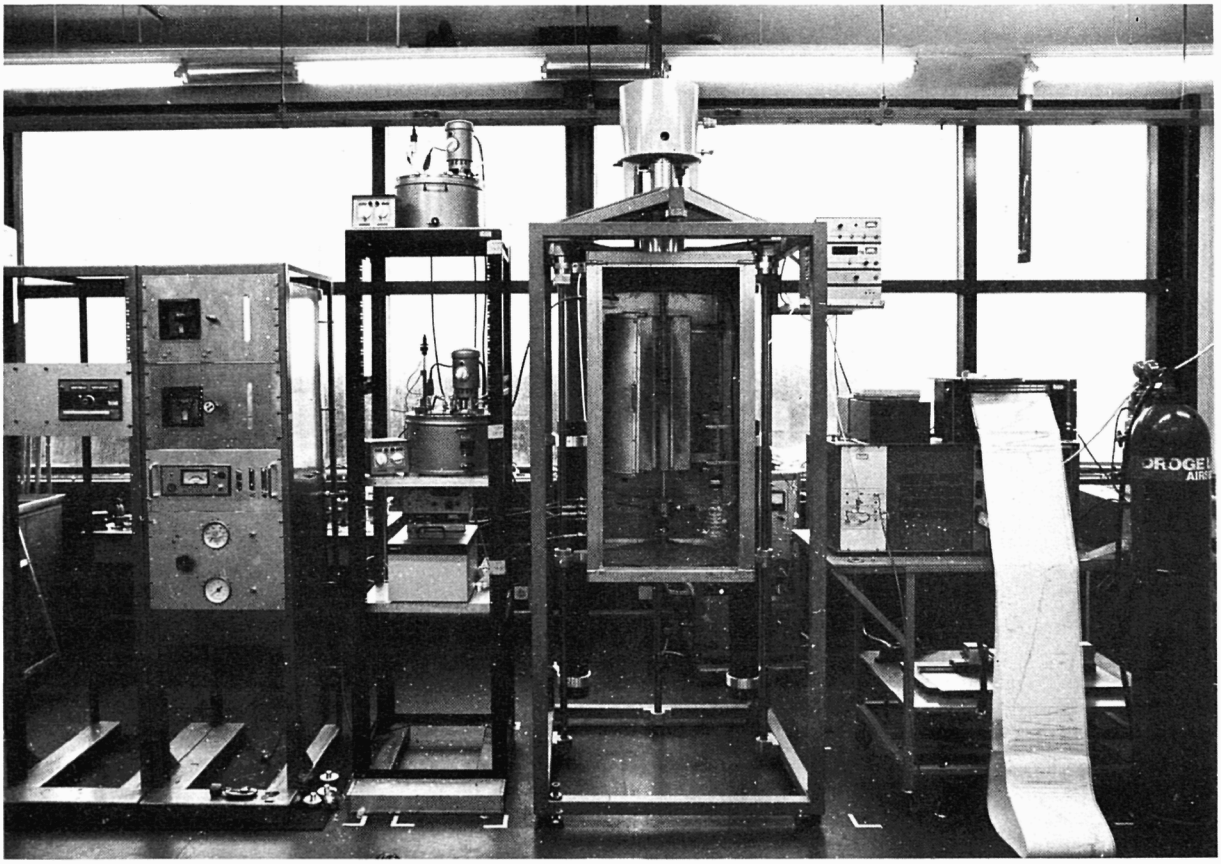


Fig. 12 Construction of test rig for corrosion investigations in carburising/oxidising environments.

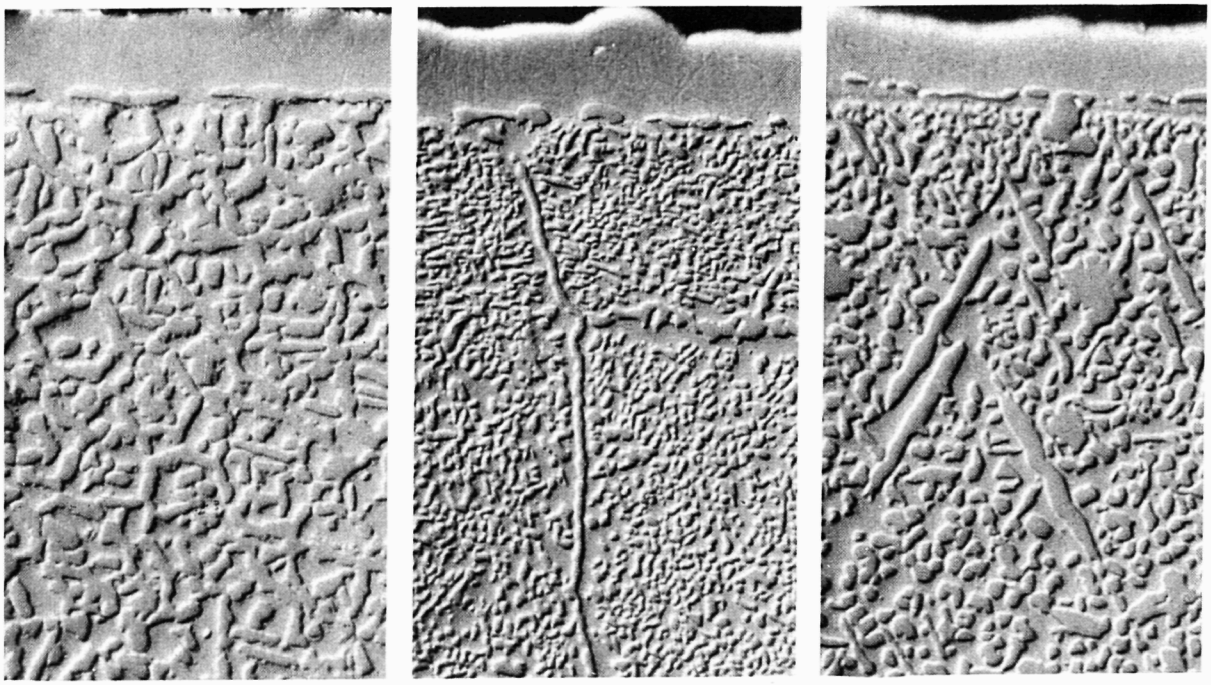
Figure 12 shows the present stage of construction of the rig designed to be used with complex gases in which both carburisation and oxidation can take place simultaneously. Continuous, isothermal gravimetric monitoring of these tests is possible using a high sensitivity thermobalance, sited directly over the corrosion chamber. 24 hours surveillance of the rig is afforded by a "SIMATIC S31" control unit which can be programmed to implement a number of actions under fault conditions including, if necessary, the controlled shutting down of the rig. The commissioning of the full installation has yet to be completed.

C. Carburisation

A further rig, designed and commissioned by HTM Petten staff, is now generating corrosion data on a range of 25^{o}/o Cr - 20^{o}/o Ni alloys exposed to hydrogen-methane gas mixtures, of high carbon activity ($a_c \pm 0.8$) at a temperature of 1000^{o}C. The kinetics of the corrosion

process are, in this case, monitored intermittently by the gravimetric examination of the interrupted specimens.

The mechanisms involved in the corrosive attack of these specimens are being studied by a range of techniques. Progressive changes in the surface appearance of each corroded specimen are being recorded by high resolution-low magnification(10x) photography. Selected specimens have been subjected to more searching surface examinations, using the Scanning Electron Microscope, Auger/ESCA and X-Ray crystallographic techniques by which the composition, morphology and distribution of the corrosive products are being determined.
Conventional cross-sectional metallography, coupled with electron probe micro analysis are enabling the extent and the mode of corrosive gas penetration and metal degradation to be established.
Fig. 13 is a series of photomicrographs taken from the examination of several 25°/o Cr - 20°/o Ni alloys exposed to the hydrogen-methane gas mixtures at 1000°C for 50 hours. It is evident that even during this short exposure period, severe carburisation has occurred, with accompanying materials degradation. Further examination of specimens exposed for periods in excess of 300 hours are indicating the change in rate of this type of attack with continuing exposure to the corrosive environment.



Wrought Type 314 *Cast HK40* *Cast Model Alloy*

Fig. 13 *Cross sectional metallography of several 25°/o chromium - 20°/o nickel alloys exposed to methane hydrogen carburising gases ($a_c \pm 0.8$) at 1000°C for 50 hours. All micrographs x 500, unetched.*

D. Surface Finish

In order to relate and compare the corrosion resistance of various alloys, certain basic test standards are essential. Adequate control over the test environment and temperature is an obvious example and has been given great consideration during the design and construction of the test rigs. In addition, surface finish (both topography and metallurgical condition) is extremely important since the rate of the initial corrosive reactions occurring at these surfaces may be radically affected by variation in this parameter. Work has been carried out in the

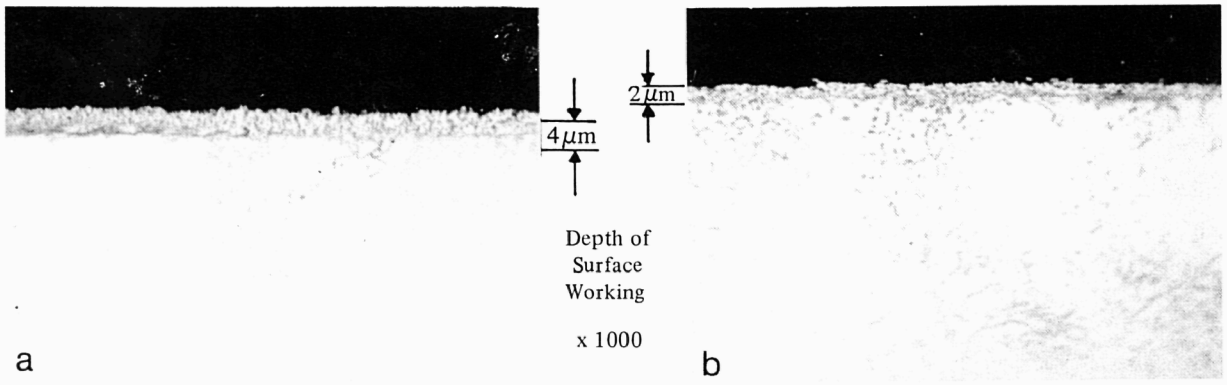


Fig. 14 *Standard surface preparation of corrosion test coupons. a) Polished with standard load on SiC paper for 20 min. b) idem for 5 min. Both etched in oxalic acid electrolyte.*

metallography laboratory in which a range of standard surfaces can be reproduced, with a known and controlled depth of surface working, as illustrated in Figure 14. In addition specimens with 'zero-working' at the surface have been prepared by electropolishing techniques prior to corrosion testing.

E. Industrial Exposure

Active liaison with Community industries has enabled materials received from relevant commerical plant, with known operational environment and conditions, to be examined. Figure 15 represents a series of photomicrographs showing the variation in corrosive attack with temperature of the internal 25°/o Cr - 20°/o Ni - 0.4°/o C (HK40) alloy pipework in a

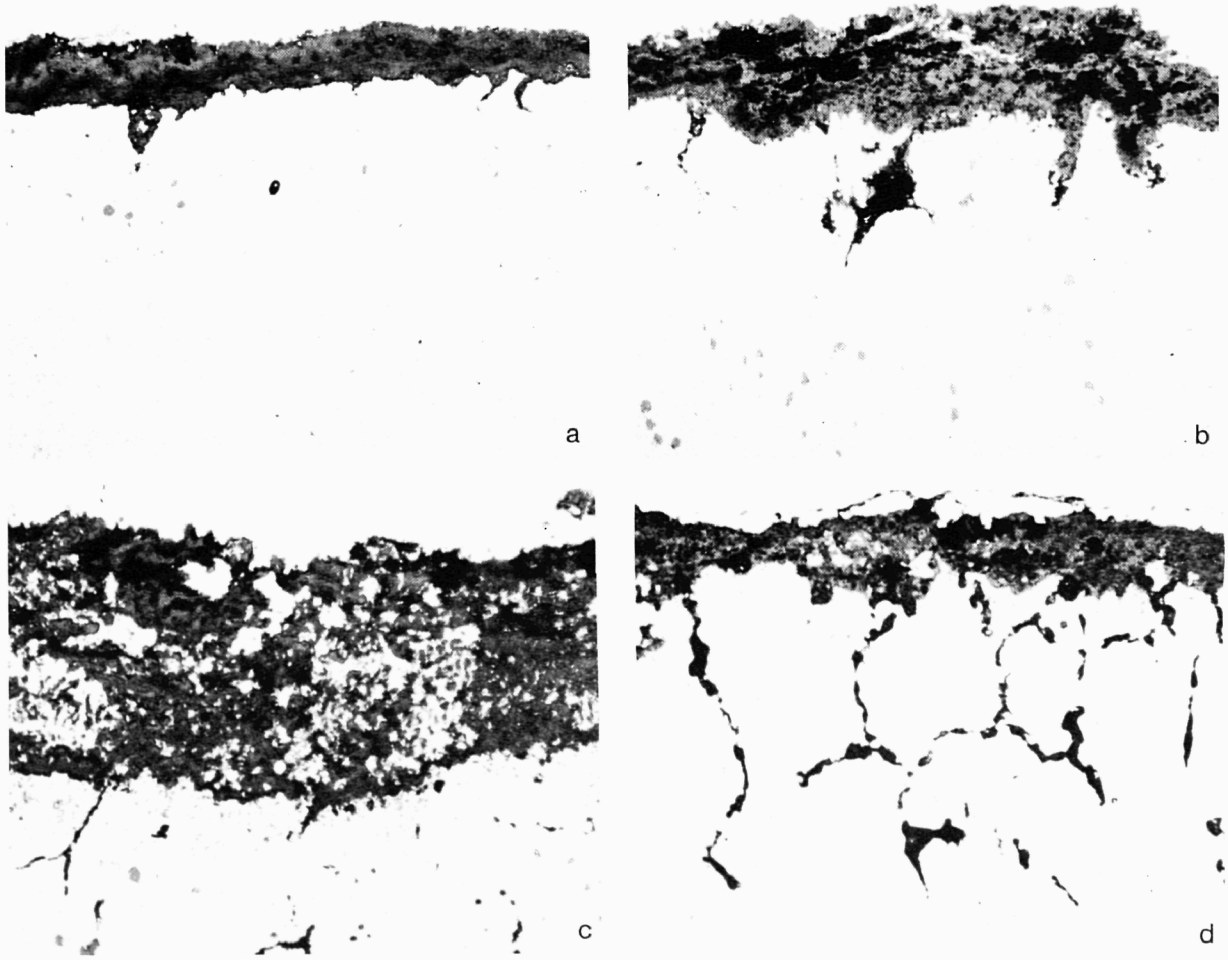


Fig. 15 *Cross section metallography of 25°/o chromium - 20°/o nickel alloy (HK40) exposed in commercial naphtha-cracking plant at ± 825°C (a), ± 875°C (b), ± 925°C (c), and ± 1000°C (d) respectively, for 22.000 hours.*

naphtha-cracking furnace. A comparison of the results of these investigations on commerically-exposed materials with laboratory-exposed test coupons is an essntial part of this research programme.

4.2 STUDIES OF THE PROPERTIES OF SCALES AND COATINGS

The aim of this work is to study oxide chemical and mechanical properties in order to allow selection of alloy/oxide systems which are capable of providing extended service in engineering applications. A programme for the structural analysis of oxide scales has been formulated in conjunction with KFR Jülich, FRG, aimed at solving the problem of hydrogen and tritium permeation in Nuclear Process Heat utilization components.

The methods available and under development for studying oxide scales fall into two categories. Firstly for the structural analysis of different alloys after exposure to the oxidising environments which are reported here, optical metallography, transmission and scanning electron microscopy, microprobe analysis and X-ray diffraction and ESCA/AUGER spectroscopy have been extensively used. Such techniques yield detailed information concerning morphology, structure and surface layer composition but unfortunately suffer from the drawback that the specimens must see a thermal cycle as the investigations are carried out at ambient temperature.
The second category of study comprises techniques which are capable of examining oxidation processes in-situ at elevated temperature.

- **In-situ study of scale formation**

One of these techniques is the vibration method (Fig. 16), for monitoring the mechanical integrity of surface layers, which has been shown for many years to be an important research tool.

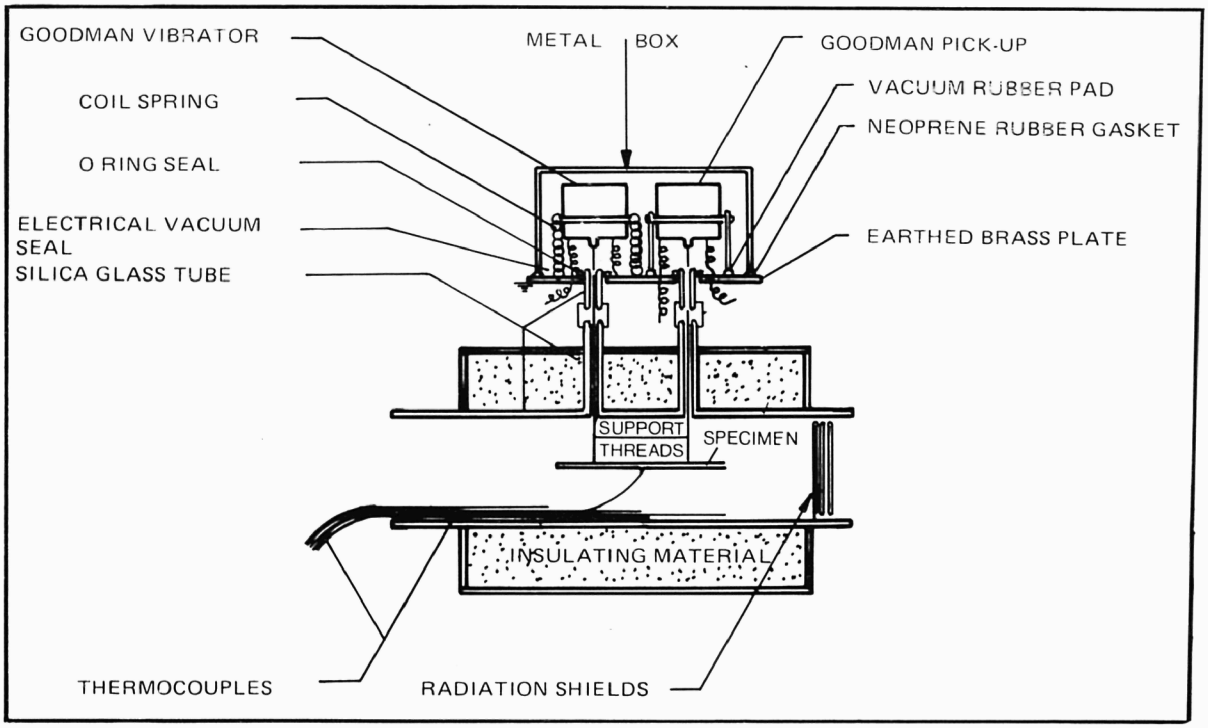


Fig. 16 Schematic diagram of vibration apparatus.

The technique relies on the measurement of resonant frequency of an oxidising metallic bar where the influence of thermal environmental changes and even superimposed loading on oxide stability can also be registered. A rig reserved for isothermal vibration tests in air has become available and has been used for preliminary testing. A major effort during the past review period was directed towards the design and construction of a rig which will enable testing by means of the vibrational technique under isothermal and thermal cycling conditions in respectively air, vacuum and toxic or potentially explosive environments.

- **Structural analysis of scaled probes.**

Samples of different candidate reformer tube alloys, Alloy 800H, Alloy 807, IN519 and IN638 have been examined after exposure in the KFA-Jülich H_2-permeation measurement rig to investigate the structure and the morphology of the respective oxide scales.

The results of the microstructural investigation can be summarised as follows:

The oxide scale on the inner (process gas) side of each tube was in all cases about five to ten times thicker than on the outer side. Since the hydrogen permeation rate is inversely proportional to the thickness of scale, the inner scale will thus induce the largest drop in permeation.

The main constituents of the inner side scales were $(CrMn)_2O_3$ and $(Cr,Mn)_3O_4$, the spinel fraction varying slightly with the alloy composition: the IN807 scale is richer in spinel, while IN519 contains less than IN638. IN519 has a discontinuous silica layer between the metal and the chromium-rich oxides. On this alloy the most uniform and adherent scale was formed.

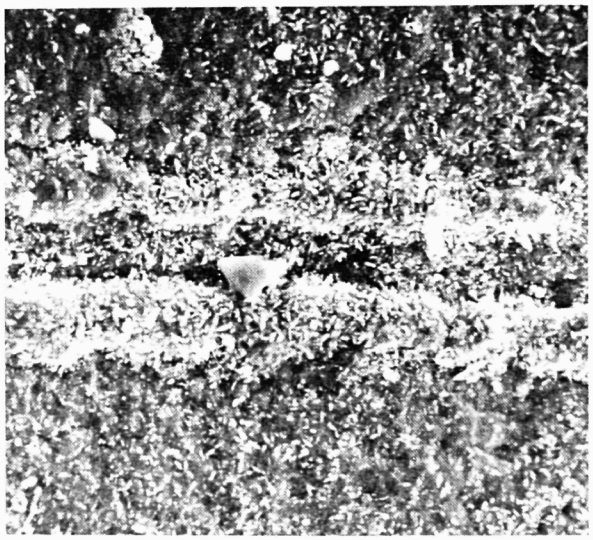


Fig. 17 SEM micrograph of scale formed on process gas side of IN519 tube.

A typical morphology is shown in Fig. 17. In an overall view the scale does not show evidence of spalling or cracking - except on the machining ridges where small hairline cracks were detected.

Alloy IN807, on the contrary, has an inner scale of variable thickness with marked signs of spalling (Fig. 18). Although the spalled areas may partially heal in the course of time, they nevertheless reduce the permeation resistance.

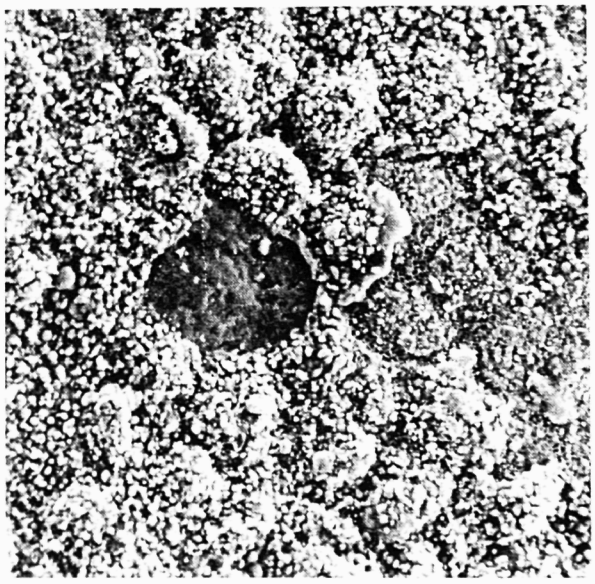


Fig. 18 SEM micrograph of scale formed on process gas side of IN807 tube.

IN638 formed a well adherent scale on the smooth surface between the machining ridges, but showed extensive spalling along these tracks (Fig. 19). This indicates a distinct influence of the surface preparation on the integrity of the scale. Fig. 24 shows other magnifications of the same scales.

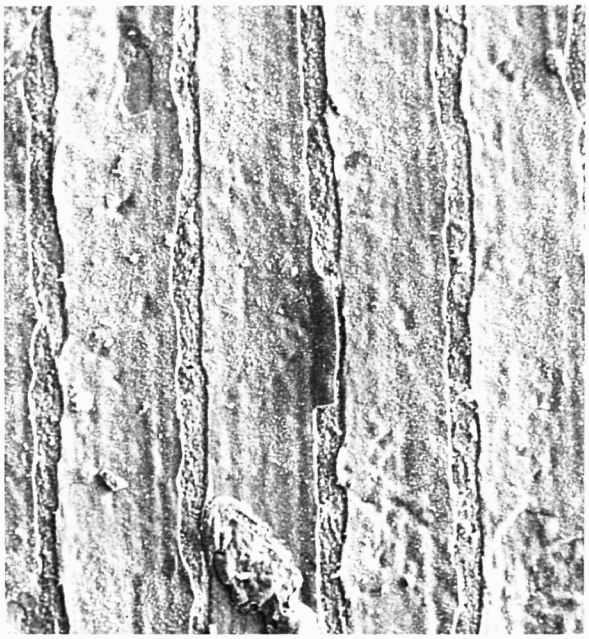
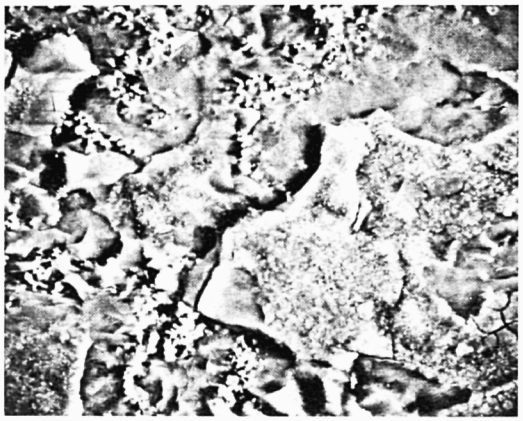


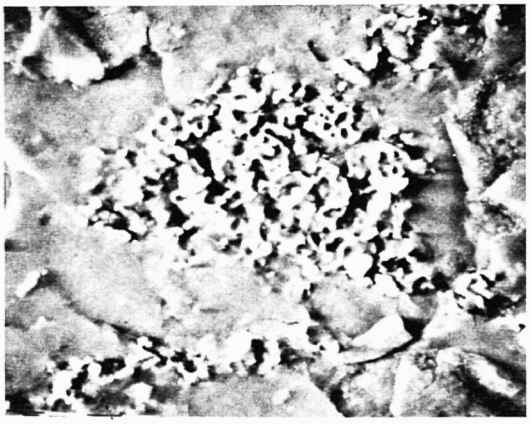
Fig. 19 *SEM micrograph of scale formed on process gas side of IN638 tube.*

In Alloy 800H an analysis of the oxides shows (Figs. 20 a-c) that three distinct scales are apparent. Transverse metallographic examination further demonstrates the complexity of the scale (Fig. 20c) where porosity can be seen as an additional factor which will essentially reduce the impermeable nature of the alloy/scale component.

Internal oxidation was confined to a zone of 20 - 30 μm near the inner wall of the IN519 and the IN638 tubes, and to 50 - 60 μm in Incoloy 807 and 800H. This phenomenon may become increasingly more important after long exposure times, even though its effects on the H_2 permeation still needs to be explored.

a





c

Fig. 20
a. *SEM micrograph of oxide, formed on Incoloy 800H.*
b. *SEM micrograph showing coral like spinel oxide formed through cracking of Cr_2O_3 rich scale on Incoloy 800H.*
c. *Section of oxide and internal oxidation formed on Incoloy 800H tube.*

5. MAJOR TEST FACILITIES

The application of materials property data to the design and development of structural components becomes increasingly difficult with the complexity of the component and the engineering application. This is particularly the case in the high temperature regime where materials react more readily with their environment, diffusion processes become an important factor, deformation is predominantly irreversible and non-linear with respect to time, and imposed stress conditions are generally unsuited to mathematical analysis. It is for this reason that organisations involved in high temperature engineering practice have a particular need to test materials in the shape and under the conditions in which they are used in service.

A considerable proportion of the research into the behaviour of high temperature materials therefore has to be carried out in environments of industrial significance. The first objective of this project is to design and install facilities for testing in such environments, taking all necessary precautions for the safety of personnel and equipment. Its second objective is to give the high-temperature materials programme an extension to the behaviour of full-size components in industrial process environments.

Accordingly the project is subdivided into 3 areas:
- Facilities for Creep and Corrosion Testing in Toxic and Explosiv Environments
- Facility for Large-Scale Component Tests (study)
- Component Testing Research

Apart from tests on components conforming to current industrial practice, there is much scope for technical research in this area. Among the most important subjects ranks research on the failure of components operating in the high temperature creep range. Such investigations are particularly needed for basic and generalized shapes like tubular components, including weldments. The studies must include the influence of practical environments upon behaviour.

5.1 THE ENVIRONMENTAL TEST LABORATORY (ETL)

The HTM programme required the installation of a battery of test rigs to investigate corrosion and mechanical property behaviour in representative environments of industrial processes. The gas mixtures involved will contain either or both toxic and explosiv components. The potentially toxic gases to be used include CO, SO_2 and H_2S and the explosiv include CO, H_2 and CH_4. All the testing machines must therefore be installed in a special controlled working area, the realization of which necessitated a particular emphasis to be placed upon safety aspects.
The construction of this "Environmental Test Laboratoy" (ETL), the supply of safety systems and the essential infrastructure was undertaken in 1977 and had reached a well advanced state

at the end of the year (Fig. 21).

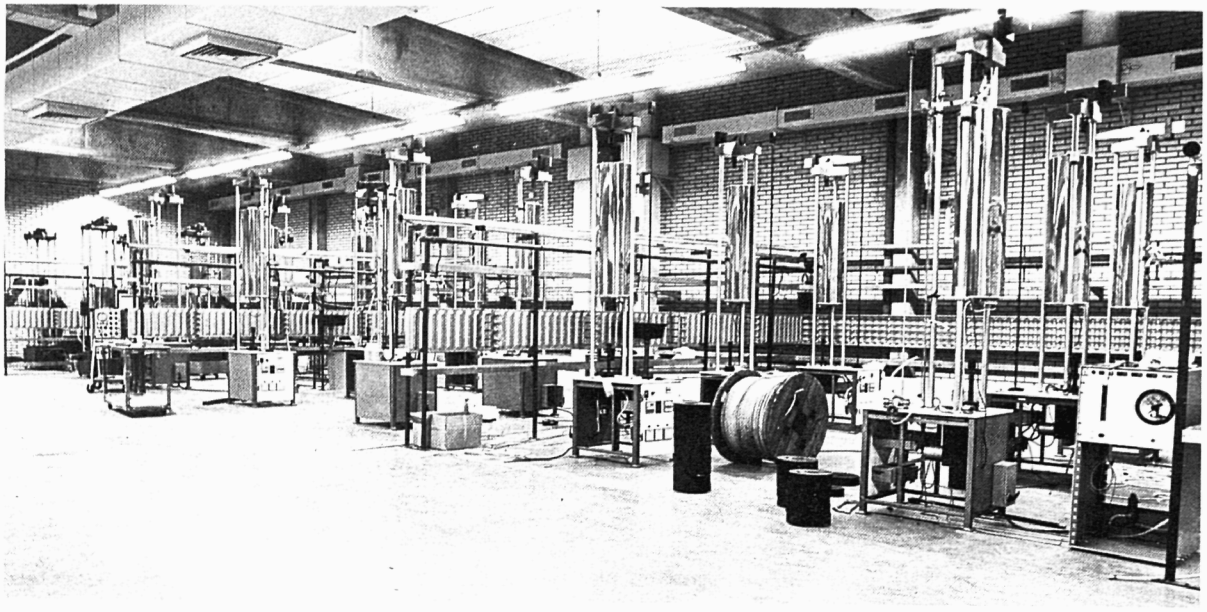


Fig. 21 The Environmental Test Laboratory in construction.

The programme of work required to realise the ETL involved site assessment, infrastructural design, safety assessment and experimental installations. For the equipping of the laboratory it was necessary to include ventilation; temperature control; laboratory cleanliness; vibration resistance, supply of test gases; cooling water and electrical power; and finally an extensive safety monitoring and data acquisition system.

A. Site

The exact siting of the new laboratory for creep and corrosion projects took safety and availability into account.
A former store area has been reconstructed and installed as the Environmental Test Laboratory. The building is divided into two areas and the larger ($600m^2$) part was chosen for housing the test installations.

B. Infrastructural Systems

The E.T.L. is presently to house some 27 creep testing machines along with the order of 13 corrosion rigs, and consequently the demand for essential services is high. Progress has in 1977 been made towards providing them as detailed below:

a) System for supply of test gases to the experimental rigs.

A gas station has been constructed outside the laboratory area to store sufficient gas bottles for the simultaneous operation of five gas lines on a grid network which has been installed throughout the laboratory. Four of these lines are to be used for "active" gas and the other line for "inert" gas.

b) Electrical power.

The installation of the electrical power network which uses an explosion proof system, has been completed.

c) Cooling water.

A pumped water grid network which, in the event of pump failure, can be supplemented

by gravity fed cooling water from reservoirs sited near the roof of the laboratory has been installed.

d) Supply of gas exhaust system.

Used gas coming from the machines has to be removed from the laboratory and this is achieved by a fan system operating on an exhaust grid which can also be powered by batteries in the event of a power shut down. The gas flow rates through the machines are very low and this is immediately diluted to negligible concentrations by air sucked into the exhaust line.

C. Safety

As far as the infrastructural design is concerned, safety requirements also necessitated the following facilities to be supplied to the laboratory:

a) Ventilation system

In order to limit the possibility of accumulation of toxic or explosive gas at a dangerous level, an extensive ventilation system is now installed which changes the ambient air (some 3 million litres in volume) every ten minutes. The ventilation system is combined with a heating system so that close control of ambient temperature can be made and it is envisaged that a cooling system must also be incorporated at a later stage.

b) Gas monitors

A system of detectors is used for assessing the levels of both toxic and explosive gases in the ambient air. These detectors are situated in the vicinity of machines and gas lines and also in the exterior gas station. They give alarms (audio and visual) when particular concentration levels are exceeded and also generate signals viable for automatic emergency shut-down procedures.

c) Safety control system

A mini-computer will be used to assimilate information concerning safety matters. It will be programmed to cause actions to safeguard personnel and equipment when necessary.

D. Experimental Installation

The creep and corrosion equipment which is to be sited in the facility is described in section 2.1, 4.1 and 4.2. This equipment has been developed to the stage that most of the units will be available for operation soon.

5.2 LARGE SCALE COMPONENT TEST FACILITY

The Petten Establishment is carrying out a study on high temperature testing techniques and facilities for structural sections and plant components. This investigation has the objective to explore the state of the art in European high temperature structural component testing, to identify the trends and future needs of this area, to develop a concept for a new advanced test facility and to investigate the feasibility of installing such a facility at Petten.
Particular attention is being paid to the type of such facilities which represent, due to their size, complexity and advanced technique, major investments and are suitable for co-operative utilization.

The trend and future need for structural tests depend on the development of the high temperature technology, and new component test facilities for the needs of the future should be realistically conceived as a function of the advanced developments. Not only new plant (e.g. hydrogen generation systems), but also new conditions in conventional systems (higher temperature, new fuel impurities) or new materials in conventional components (ceramics for metals) create new requirements.
To take into account the available state of knowledge as well as the future needs, the study was subdivided into 2 special investigations, one to inventorize relevant high-temperature component test facilities existing in Europe and the other to study the need trends in order to suggest in a preliminary fashion some alternative areas for further consideration. During the year 1977, therefore, the study was organized as a co-operation between JRC Petten (on the first aspect) and the Battelle-Institute, Frankfurt/Main (on the second).
The Battelle-Institute has made for their basic orientation some expert interviews to investigate the opinions of relevant organizations interested in this field. These represented a cross section of the industrial interest in high-temperature technology. Parallel to these interviews, information was collected from a literature survey, expert meetings and conferences.

General problem areas for which technological solutions require or suggest component tests can be summarized as follows:

- The influence of fabrication parameters, material structure and component geometry upon the strength in service (ceramics components in particular).
- Strength of components, shape and size influence under service conditions, improvement of design and life prediction, reduction of safety factors (gas turbines in particular).
- Effect of various material defects and structural conditions upon the component behaviour under service load (general).
- Strength in service of welds in tubes, tubular components, transitions, intersections etc. (steam reformers and crackers in particular).
- Non-destructive methods and their development, remnant life prediction and testing (steam reformers and crackers in particular).

Apart from industrial requirements, there are also requirements for such tests in the interest of licensing, supervision and inspection authorities, research organizations and technical assurance companies. These requirements are in general related to safety aspects, which have similar problem areas as those above.

The Petten inquiry on European test facilities has yielded an inventory of about 70 facilities which are classified by environments:

Burning fuel oil; gas or coal, coal gasification; helium; liquid sodium; carbon dioxide; steam; reformer gas; air; argon; vacuum.

The knowledge of the technological problem areas for which high temperature component tests are needed, is now rather well established, and the next time will be used for a deeper evaluation of these areas. The ultimate goal of the study is the specification of a large scale test facility as basis for a decision on its potential realization. It is planned that this technical specification and a resource planning will be produced in 1978.

6. STRUCTURAL ANALYSIS FACILITIES

The structural analysis services supply broad analytical support to the R & D projects described in sections 2, 3 and 4. The most important scientific objective for the materials laboratory is the proper characterisation of materials structures in a very general sense.
Whereas during 1976 existing equipment has been reconditioned and some new instruments have been installed, during 1977 new techniques have been developed, existing equipment has been extended and obsolete instruments have been replaced.

6.1 METALLOGRAPHY

The main tendency was to complete and develop new techniques for phase identification. The "Pepperhoff" coating unit, which permits the identification of alloy phases in-situ has been completed. Some typical photos are shown in Figures 22 and 23 (here reproduced black-white).

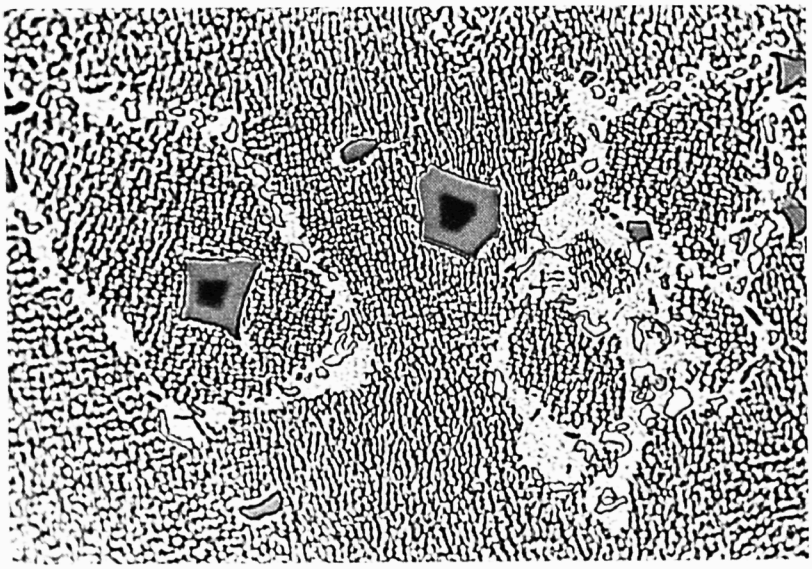


Fig. 22
Segregation of Ni_3Al/Ti (γ') to grain boundaries in IN100 following creep testing at 950°C/100h.

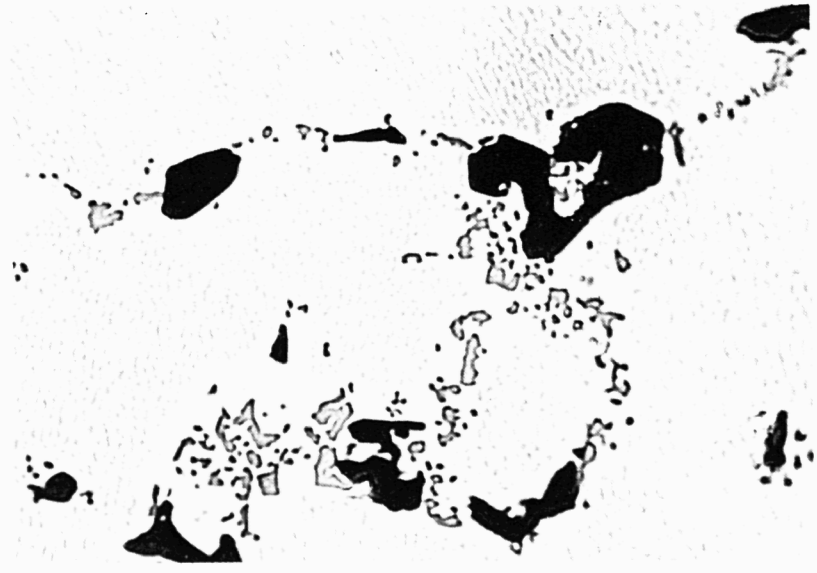


Fig. 23
Distribution of carbides (TiC and $Cr_{23}C_6$) in creep tested (950°C/100 h) IN100.

Another method which is useful for characterisation of carburised steels, the potentiostatic etching, is being developed for the special needs of this service.

6.2 SCANNING ELECTRON MICROSCOPY

In the electron microprobe laboratory an X-Ray energy spectrometer has been added to the existing wavelength dispersive spectrometers. This spectrometer is used for fast quantitative chemical analysis.
Used as a scanning electron microscope, the instrument is giving excellent service in the study of fracture surfaces of creep specimens and of scales due to high temperature corrosion (Figures 24 a-d).

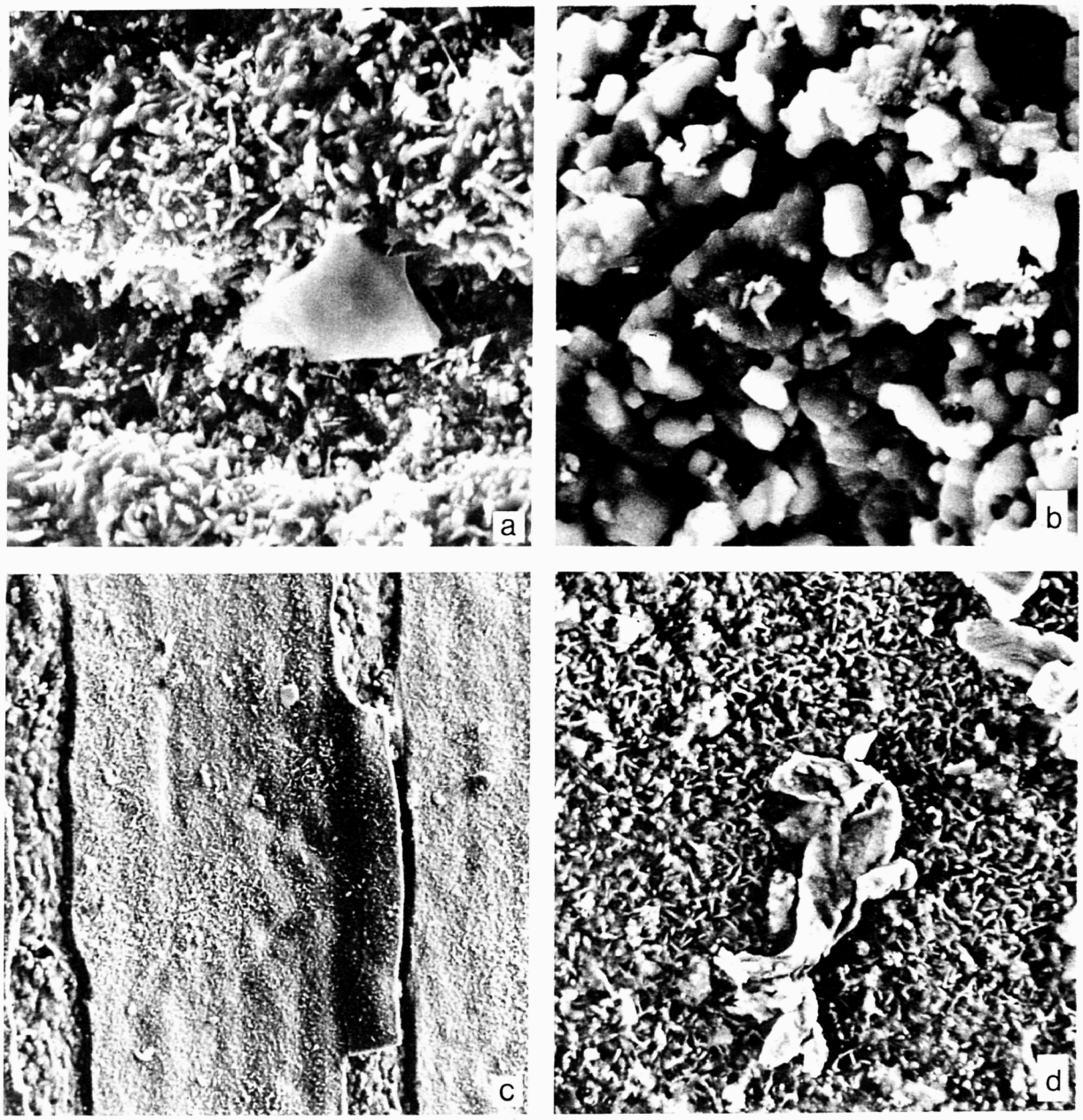


Fig. 24 *Morphology of the oxide scale formed in a simulated reformer gas.*
a. on a IN519 alloy (2.000x) *b. on a IN807 alloy (3.300x)*
c. on a IN638 alloy (300x) *d. on a IN638 alloy (1.000x)*

6.3 TRANSMISSION ELECTRON MICROSCOPY

An energy dispersive spectrometer (EDS) has been interfaced with the Philips EM300 transmission electron microscope. The combination of the EM300, EDS and the attachment for scanning transmission electron microscopy (STEM-unit), makes the electron microscopy a powerful analytical instrument, capable of solving various metallurgical problems, such as the examples shown in Figure 25 and Figure 26.

In the STEM mode a narrow electron beam scan of a specimen permits the analysis of precipitates down to some 500 Å in size.

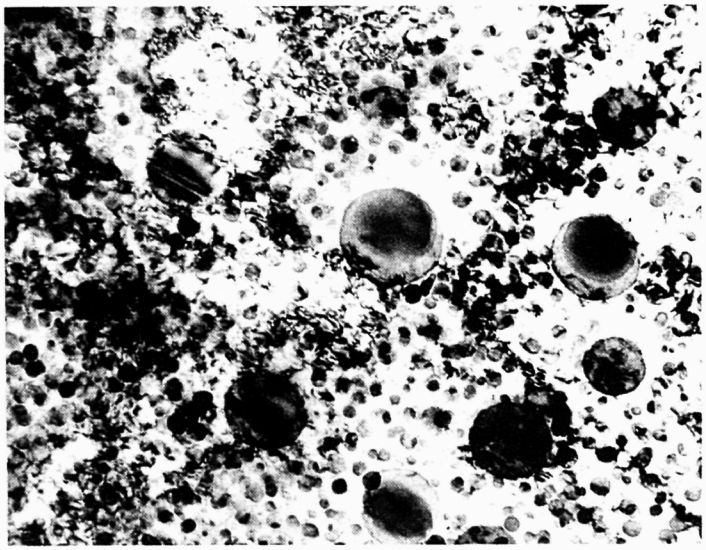


Fig. 25 *Microstructure of Waspalloy, showing two families of γ′ particles (50.000x).*

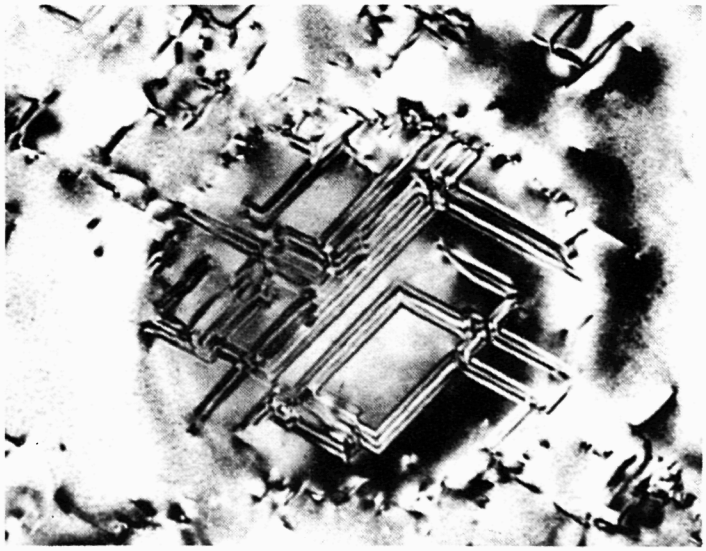


Fig. 26 *Detail of γ′ particle showing planar defects (200.000x).*

6.4 X-RAY LABORATORY

The new Guinin camera based on the Seemann-Bohlin focussing principle is now fully operational. It proved to be an extremely powerful tool for the identification of phases, which is one of the main tasks of the X-ray laboratory. The background reduction through the use of highly monochromatic CuKα 1 or CrKα 1 radiation permits the detection of very scarce phases. The focussing geometry in addition minimizes the line broadening, so that interplaner spacings differing as little as 0.2 $^{o}/_{o}$ can still be easily resolved. The computer programmes for calibration and calculation of lattice spacings have been specially designed in order to accommodate future semiautomatic measurements on other instruments as well.

ORGANIC MATERIALS

TABLE OF CONTENTS

Introduction

The Organic Materials Laboratory has continued its activities in 1977, as a unit for research and development in the sector of organic chemistry in the frame of the JRC programme on "Standards and Reference Substances".
For the 1977/80 period the programme has been renamed "Measurements, Standards and Reference Techniques" (METRE), subdivided into the following five projects:

- Project 1 : measurements of nuclear data,
- Project 2 : nuclear reference materials and techniques,
- Project 3 : non-nuclear reference materials and techniques,
- Project 4 : scientific support to the Services of the Commission,
- Project 5 : scientific support to the Secretariat of the Community Bureau of Reference (BCR).

The programme is carried out in the JRC Establishments of Ispra, Geel and Petten, with JRC Petten contributing to project 3, 4 and 5. The principal aim of the work at JRC Petten is to assist the harmonization and calibration of analytical techniques within the Community in the fields of organochemical products (Petroleum products, plastics and rubbers, environmental specimens, cosmetics, foodstuffs, e.g.). In this context, special importance is attached to the development of reference materials in accordance with the Council directives on organic products and environmental aspects.

1. PROJECT 3: NON-NUCLEAR REFERENCE MATERIALS AND TECHNIQUES.

1.1 POLYAROMATIC HYDROCARBONS

The experimental work undertaken aims at the preparation and certification of a set of very pure polyaromatic hydrocarbons (PAH; purity 99%o or better) which have to serve as reference and calibration materials for the analysis of hazardous and carcinogenic PAH's in a wide range of environmental samples and industrial products. The analysis of these compounds has to be performed by at least three independent techniques such as gas-liquid chromatography (GLC), high performance liquid chromatography (HPLC), UV/fluorescene spectrophotometry, mass-spectrometry (MS) or differential scanning calorimetry (DSC).
In order to test the potential of the various analytical techniques proposed for the experimental work and to get an indication of the reproducibility limits which can be achieved, a pilot ring analysis has been organized by JRC Petten on a commercial pyrene, with a quoted purity grade of better than 99%o. The analytical results which have been obtained on this PAH-compound by means of GLC, HPLC, GC/MS, UV and DSC are summarized in Table 1.
Following the pilot analysis, the purity of three synthesized PAH's (benzo(b) and benzo(k) fluoranthene, benzo(b) chrysene) has been analysed using the same analytical techniques. The results of these analyses are summarized in Table 2. It can be seen that the materials are of very high purity. In fact, it was impossible to detect any impurities by conventional techniques. Only by a specially adapted MS-technique (direct injection, low ionisation

Table 1 : Pilot analysis of pyrene

Method	Pyrene (°/o)	Impurities		
		1	2	3
GLC	97.9	1.8	0.18	0.13
	97.8	1.7	0.15	0.1
HPLC	98.0	1.75		
UV	(98.3)	1.7		
DSC	96.8			
Average (without DSC)	98.0 ± 0.2			
Identification		4,5-thiophenanthrene	dihydropyrene	fluoranthene

Table 2 : Analytical results for three PAH-candidate reference materials.

PAH	benzo (b) fluoranthene	benzo (k) fluoranthene	benzo (b) chrysene
GLC	99,9	99,9	99,9
HPLC	99,9	99,9	99,9
MS	99,9	99,9	99,9

potential) could an impurity be detected in one of the PAH candiate reference materials (benzo (k) fluoranthene) at a concentration level of < 0.1°/o. Therefore it is expected that these three PAH-materials can be certified and distributed as reference materials by the Community Bureau of Reference (BCR) in 1978.

1.2 FLASHPOINT DETERMINATION

The certification measurements on five hydrocarbon materials (n-octane, p-xylene, n-nonane, n-decane and n-undecane) which are intended to be used as reference materials for flashpoint determination have been completed in 1977. The results of all the participating laboratories have been collected, corrected for barometric pressure and statistically analysed.

As a result of the inter-laboratory round robin measurements the following values have been proposed for certification:

Table 3 : Proposed certified flashpoint temperatures.

Material	Flashpoint temp. (°C)	Standard deviation (°C) (for reproducibility) S_R	(for repeatability) S_r
n-Octane	14.0	0.8	0.6
p-Xylene	26.0	0.7	0.5
n-Nonane	32.0	0.9	0.6
n-Decane	49.0	1.1	0.8
n-Undecane	63.0	1.2	1.0

These results have been obtained with equilibrium methods (IP 303/IP 304) *), using the Setaflash, Abel, Abel-Pensky, Pensky-Martens and Tag apparatus (closed cup). No statistically significant variations were observed between the results of the various standard flashpoint apparatus cups.
A certification report has been prepared **) and the proposed flashpoint temperatures have been certified officially.

1.3 ATMOSPHERIC DUSTS

The objective is to prepare a reference material for the analysis of hazardous components (toxic metals like Pb, Hg, As, etc. and organic traces) in environmental dust samples. To this end, a sizable amount (about 25 kg) of a homogenized fly-ash with a particle size smaller than 10 μm will be analyzed for the elements and components of primary importance (Pb, Cd, Hg, Ca, Co, Cu, Mn, Na, V, Zn, F and extractable organic fraction). The Petten laboratory is to participate mainly in the analysis of organic trace constituents.
For the analysis of organic components, a combination of gas chromatography / mass spectrometry is the principal tool with gas, liquid and thin layer chromatography as back-up methods. Total organic content is determined by solvent extraction and gravimetry.
Since the final homogenised fly-ash material was not available, the work was limited to the analysis of an advance sample of the fly-ash with the intention of assessing the concentration ranges which can be expected for this material. An important result of this analysis was the finding that the organic content of the fly-ash specimen was conspicuously lower than originally expected (less than 0.1 instead of $>$ 1 wt.^{o}/o). Therefore, the proposed quantitative determination of individual toxic organic components (especially cancerogenic polyaromatic hydrocarbons such as benzo(a) and benzo(e)pyrene, indeno(1,2,3-c,d)pyrene, benzo(g,h,i)perylene and similar substances) seemed to be inadvisable because of the very low concentrations. In deliberation with the BCR working group "Environmental Dusts" it was therefore decided to limit the analytical work on organic fractions of the fly-ash material to a quantitative determination of total content and to a qualitative identification of the main constituents.

1.4 POLYMER ADDITIVES TO LUBRICATION OILS

Two commericially available samples of polyisobutene described as low molecular weight have been obtained to allow a preliminary investigation of experimental conditions suitable for more detailed study. Both samples have been examined by dilute solution viscometry in three solvents at 30^{o}C: toluene, cyclohexane and carbon tetrachloride.
Cyclohexane gave the most repeatable results but neither polymer can be considered as a candidate reference material as the molecular weights were found to be too high ($>$ 350.000 daltons for the "low" molecular weight sample and $>$ 1.300.000 daltons for the "medium" molecular weight sample). A material with a viscometric molecular weight in the range 10 - 50.000 daltons is considered ideal and samples in this region have been requested.

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*) *IP Standards for Petroleum and its Products, 33th edition (1976): - IP 343/74 rapid tests for flashpoint - IP 304/74 flash tests using the cup of any standard closed cup apparatus.*

**) *D. Lewis, L. Haemers and W. Karcher: The certification of five hydrocarbon materials for the determination of flashpoint (temperature range 10 to 65^{o}C) (EUR-report, in print).*

Following the proposed certification of a sample of polystyrene as a viscometric standard and the proposal to use the same material as a GPC (gel permeation chromatography) standard, this laboratory will take an active role in the practical work and will evaluate the sample as an alternative polyisobutene as the sought after viscometric standard for lubrication oil additive if the latter should be unobtainable with the described molecular characteristics.

1.5 FOOD PACKAGING MATERIALS

Over the last decades the sale of packaged food products has increased very much in the member countries of the European Community and more recently it has been recognized, that the migration of additives from the packaging into food may present health hazards for the consumer. Therefore, legislation has been passed in various countries in order to set limits for the migration of hazardous packaging additives into foods. Also, the ministers Council of the EEC has issued a directive on this matter.
The implementation and control of these limitations require basic data of migration rates of additives in polymers and the development of suitable analytical techniques and calibration materials.

In a first approach a comprehensive literature survey on this subject has been carried out (shortly to be published as a EUR-report*). The main conclusion to be drawn from this report was the underlying need for standardisation in all areas of this field of research.
Secondly, preliminary work has been undertaken to produce cells for the study of the diffusion/migration phenomenon. Two cells have been developed and it is now possible to follow gaseous diffusion as well as that occurring when foodstuffs and plastics are in contact. In the first case noble gases have been employed to calibrate the instruments; a diffusion cell linked directly to a small quadrupole mass spectrometer, and a typical diffusion coefficient of 3.32×10^{-10}cm/s has been found for the diffusion of argon through a commercial low density polyethylene film. This was in good agreement with literature values for similar inert species **). In the second instance a cell based on the model produced by Figge et al***) has been produced to study the movement of plastics additives through a plastic film. The development of this cell is detailed in Technical Note P/09/77/45 ◙). At this moment work is in progress to obtain data from these cells and both chloroform and carbon tetrachloride are being employed as food simulants.
Some work has been carried out on the migration of plastic additives into the fat simulant HB307 too.

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*) *Migration phenomena in food packaging (A literature survey), EUR 5979 MF (in press).*
**) *Rubber handbook, Section III, Permeability Coefficients.*
***) *Deutsche Lebensmittel-Rundschau, 67 (1), 9, 1971.*
◙) *Technical Note P/09/77/45, A Schwarze, G. Haesen, E. Nagy.*

2. METRE-PROJECT 4 : SUPPORT TO THE SERVICES OF THE COMMISSION

Experimental work in support of the Services of the Commission has been continued in the organic products sector. In particular, the Petten Laboratory facilities have been used for developing and testing analytical methods for colouring agents in cosmetics in support of the Service for Environmental and Consumer Protection and for tariffication purposes in support of the Common Customs Tariff (CCT).

2.1 METHODS OF ANALYSIS OF COSMETIC PRODUCTS

Work on the analysis of permitted colouring agents in cosmetics has continued in 1977. Samples of the constituent colorants have been assayed and distributed as working reference materials. A combination of analytical techniques was used for this assay including TLC, HPLC and solution spectroscopy. The results are shown in Table 4.

Table 4 : Reference assayed colorant samples.

C.I. number	Names	Cosmetics directive restrictions	Reference sample purity
12085	Pigment Red 4 D & C Red Nr. 36	not more than 3°/o	97.8°/o pure colour
15630	Pigment Red 49 D & C Red Nrs. 10-13	not more than 3°/o	67 °/o pure colour
45370	Fluorescein D & C Yellow Nrs. 7,8	not more than 6°/o	98.3°/o pure colour
45396	Solvent Orange 16 Dinitrofluorescein	Acid form only at not more than 1°/o in lipsticks	98°/o pure colour

For the lipstick formulations, three methods of analysis have been compared and of these one method, employing complete separation of all ten colorants present by liquid-liquid partitioning followed by TLC, has been shown to be more reliable than the others. Further samples of cosmetics of other product-types (i.e. not lipsticks) have been obtained from the cosmetics industry and modified methods of analysis will be evaluated to determine the constituent colorants. In this context, HPLC and TLC have proved effective in the direct analysis of a foaming bath product containing fluorescein.

2.2 SUPPORT TO ADMINISTRATION OF CCT.

Classification of polymer additives.

The Organic Labory has taken part in a circular analysis of a set of selected industrial products (polymer additives in lubricating oils) with the aim to test and define suitable analytical

methods for classifying these products (CCT headings 27.10 and 39.02 respectively). The results of the measurements carried out for the CCT-administration are described in detail in a Technical Note*).

The reproducibility limits of the vacuum distillation results have been tested by varying the team of operators systematically (3 different teams). In all cases, the standard deviation of distillation values was seen to be below $\pm$ 5°C, which is well in line with the precision specifications of the ASTM-method, which allows for variations of $\pm$ 5°C for the same operator and between $\pm$ 8°C and $\pm$16°C (depending on the distillation temperature) for different teams of operators and laboratories. A final report, covering all the experimental results obtained has been issued to the Administration of the CCT*).

Determination of milk fat in presence of vegetable fat.

For the correct tariff classification of certain food products a suitable analytical method is required since it has become apparent that the conventional method which has been in use can lead to an incorrect classification. To this end, experimental work has been started with the purpose of testing and adapting a gas chromatographic method**) for the determination of milk fat in food products in presence of other fats in order to permit a clear distinction to be made between the respective tariff classes (19 08 B II d1/d2).

*) *Distinction between low-polymerised hydrocarbons of heading 27.10 and 39.02 of the CCT. Technical Note P/09/77/5.*

**) *Cristopherson, J. Dairy Science 52, 1289, 1969.*

3. METRE-PROJECT 5 : SCIENTIFIC SUPPORT TO BCR SECRETARIAT

In support of the secretariat of the Community Bureau of Reference (BCR), JRC Petten has supplied the scientific secretaries for the three working sub-groups which are active in the organic sector and for some of the corresponding groups of specialists.

3.1 PETROLEUM AND RELATED PRODUCTS.

In this working sub-group, the work on three project items ("RM for flashpoint", "RM for PAH-analysis" and "RM for metal traces in synthetic protein") has been continued or completed under the guidance of the corresponding specialist groups. The circular analysis for flashpoint reference materials has been successfully completed and a certification report has been made available.
On the first three PAH candidate reference materials, the majority of the results of the certification analysis programme has been obtained. The samples for the certification analysis of metal traces in synthetic protein have been distributed after homogeneity of the material had been confirmed and the first analytical results have become available.

Another reference materials project ("RM for sulphur determination in gas and fuel oils") is in a preparatory stage and discussions have commenced on the feasibility of reference materials for fuels and lubricants.

3.2 PLASTICS AND RUBBERS

Under guidance of this working subgroup an intercomparison exercise on polystyrene viscometry has been concluded and the candidate reference material for molecular weight measurements of poly (vinyl/acetate) has been prepared and analysed. Based on the results of the viscometry intercomparison exercise on polystyrene, an activity has been started to certify the molecular weight distribution of this polystyrene material by means of gel permeation chromatography.

3.3 ORGANIC ANALYSIS

Within the group of specialists "Organic elemental analysis" which resides under the WSG "Organic analysis", the certification analysis has been completed and a draft certification report has been prepared. Work on three more candidate reference materials for elemental analysis is in progress.
A reference materials project for an organic compound containing several heteroelements together (Br, Cl, F, N) has been started in the second half of 1977.

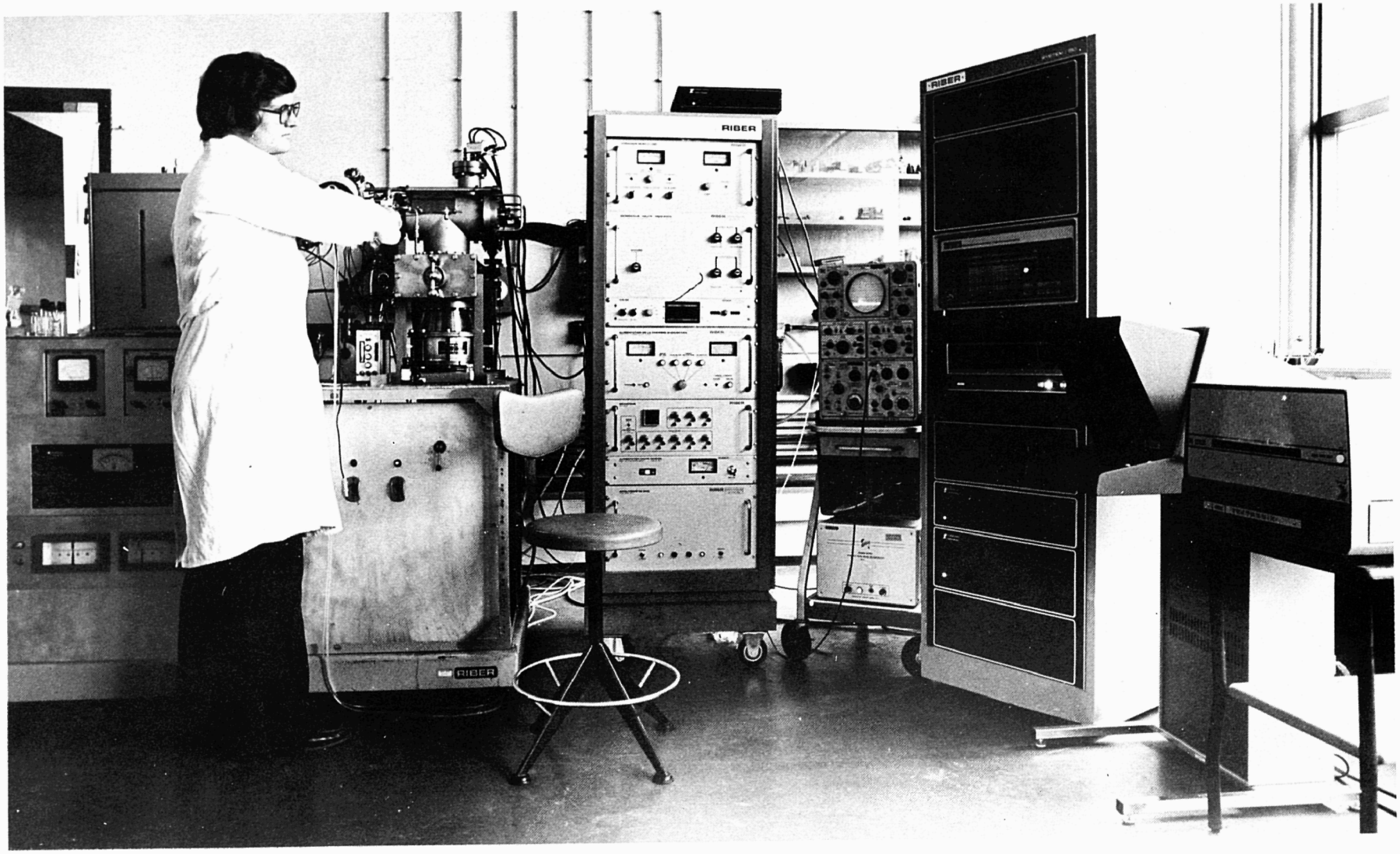

Fig. 1 *Combined gas chromatograph and quadrupole mass spectrometer with interactive computer control and data reduction system.*

III GENERAL SERVICES

INTRODUCTION

To maintain the Establishment's scientific activities at a reasonable level of efficiency, a series of supporting actions are necessary to provide both for the material and social needs of the staff and for the provision of general financial supply, scientific, technical and information services to the research programmes.

For the convenience of this report these affairs are all grouped together and reported in this chapter.

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1. ADMINISTRATION, PERSONNEL

The staff authorized for 1977 totalled 165, of which 161 were present at the end of the year. In the course of 1977, 7 agents left the Establishment (by death, transfer to another Establishment, or resignation and retirement). 7 new staff members were recruited as temporary agents.
The number of auxiliary staff totalled 4. A further 14 students and research fellows working in the frame of irradiation technology and materials science, stayed at Petten, most of them for the preparation of their doctorate.

The distribution of the personnel by programme and by category (scientific and administrative) was as follows (situation on 31.12.1977):

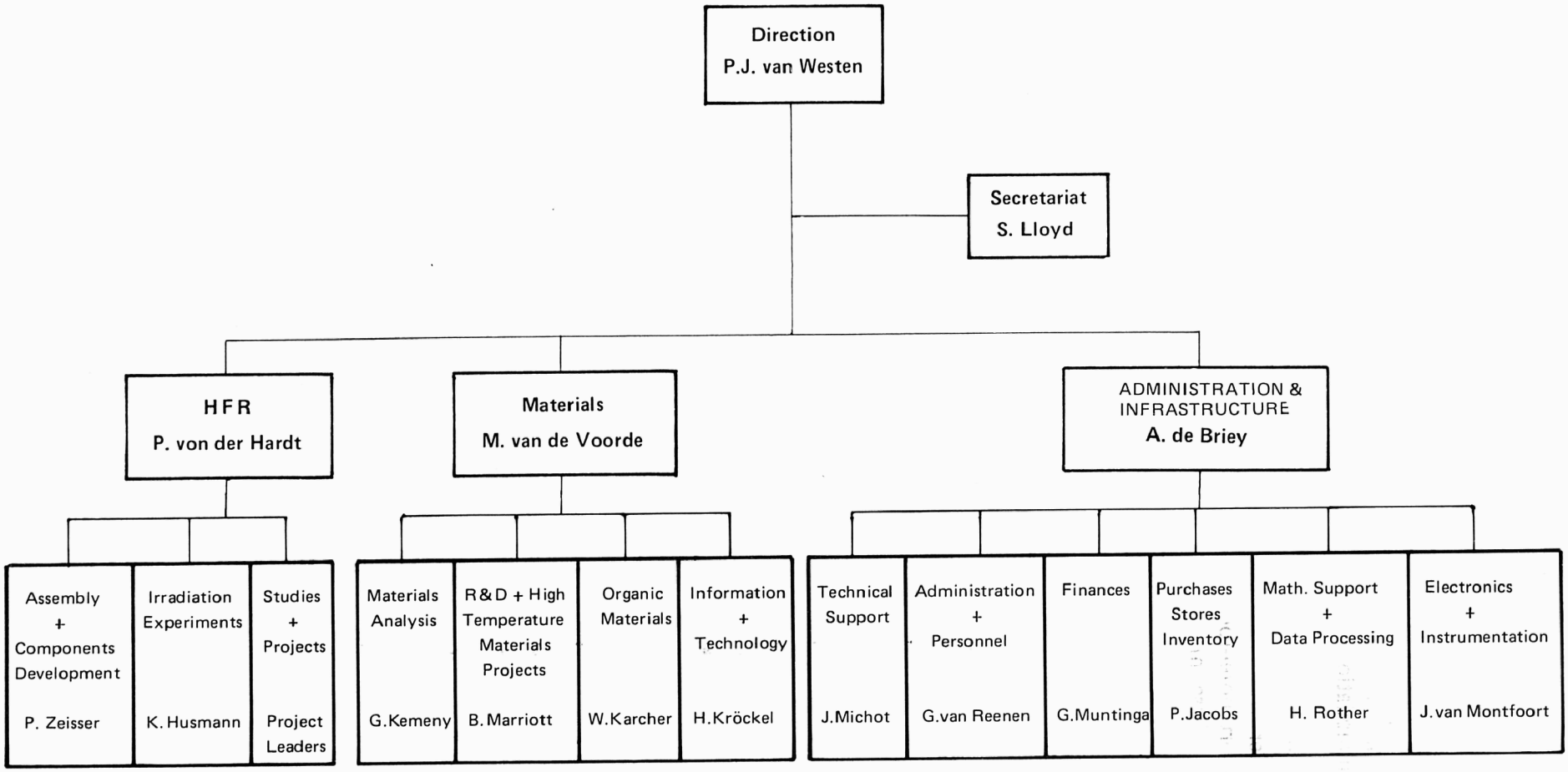
Programme	Scientific staff	Staff for technical support	Staff for general and administrative support *	Totals
HFR	42	27	23	92
High Temperature Materials	34	4	11	49
Organic Materials	13	2	5	20
Totals	89	33	39	161

* Directorate, administration, finance, infrastructure and administrative support to the scientific divisions.

The distribution of the personnel by nationality is stated in the next summary:

Category	German	British	Belgian	French	Italian	Luxemb.	Irish	Dutch	Total
A	14	8	8	2	--	--	--	3	35
B	25	7	16	4	5	2	2	12	73
C	4	--	3	3	2	--	--	35	47
D	--	--	--	--	--	--	--	6	6
General Totals	43	15	27	9	7	2	2	56	161

FUNCTIONAL ORGANISATION CHART OF THE PETTEN ESTABLISHMENT



Because of the shortage of dwellings in this region of the Netherlands, the Administration of the Establishment has to take care of the housing problems of the staff. For that purpose, the negotiation and the guarantee of rent for more than 100 houses and appartments is handled by the Administration. Besides this, the Administration rents 11 appartments and 1 house, putting them at the temporary disposal of new coming staff and students as furnished accommodation.

The number of conferences, symposia and meetings held at the Petten Establishment, has considerably increased in the last few years. During 1977, the Administration has organised transport, hotel-rooms, meeting- and conference rooms, simultaneous translation etc. for about 20 of these meetings.

2. FINANCE

In 1977 the total costs for the Petten Establishment amounted to 12.265 k.U.A (commitments).
The distribution of this amount between the three programmes executed at the Petten Establishment was:

- High Flux Reactor (HFR)	9280
- High Temperature Materials (HTM)	2250
- Standard and Reference Materials (METRE)	735
Total	12.265

The HFR programme was financed by two member states as a J.R.C. supplementary programme, the two other activities belonged to the common programme.
The breakdown of these amounts by character of the costs is as follows:

	H.F.R.	H.T.M.	METRE	Totals
- staff costs	2431	1446	478	4355
- general establishment running costs	732	321	102	1155
- site and building maintenance	79	31	10	120
- general technical services	85	25	4	114
- small investments	263	286	80	629
- technical materials and equipment maintenance costs	421	141	61	623
- HFR exploitation:				
- fuel cycle	1524	--	--	1524
- daily operation	3644	--	--	3624
- electricity	245	--	--	245
- insurances, miscellaneous	56	--	--	56
Subtotals	9481	2250	735	12.465
Incomes	200	--	--	200
Totals	9280	2250	735	12.265

Beyond these costs users of the HFR contributed direct by 643 k.U.A to the costs of irradiation projects executed on their behalf.

3. SUPPLY

3.1 Purchasing

Almost 1700 purchases have been handled during the year, representing to a sum of 2,859 M.U.C. Included in this are dossiers prepared for large items, where a procedure of asking for tenders and subsequent examination by an official advisory committee (CCAM-CCR), is followed before authorisation is given.
A particular problem has been the effects on research programme planning of delivery dates not being maintained by manyfacturers, often due to the developmental state of advanced scientific equipment.

3.2 Stores

1977 has seen the continuation of the reorganisation of the central stores and its stock.
The materials and objects held there are under continuous review by a users' committee and by the end of 1978 it is anticipated that the work of updating and reclassification will have been brought up to date.

4. INFRASTRUCTURE

During the reporting period, a considerable effort has been applied to studies and preliminary planning to improve the physical protection of fissile materials held on the site, in line with the International Atomic Energy Agency recommendation IAEA-225. By the end of the year a number of schemes have been considered and alternatives selected for further examination.

The technology hall has been completely re-painted during the year and a special room constructed to house an electron-beam welding equipment to be installed early in 1978.

An underground store has been provided for the storage of toxic or inflammable organic chemicals, prepared and certified at Petten as "reference substances" in connection with the "Community Bureau of Reference". Construction followed the "cut and fill" method using a prefabricated concrete garage as the inner liner.

As described in section 5.1 of the High Temperature Materials part, a small hangar, formerly used for the storage of bulky items of equipment, has been converted for use as a laboratory of 600 m^2 where long term mechanical testing in toxic and explosive gases can be carried out. By the end of the year the ventilation and heating systems have been installed and special electrical and cooling water supplies provided. Installation of piping to supply and exhaust the gas atmospheres used in the experiments has begun and a multi-pair instrumentation cable laid to the Materials Laboratory building.

In addition to these particular items, normal maintenance and construction work has been carried out on the site and buildings.

5. SCIENTIFIC AND TECHNICAL SUPPORT

5.1 Library, Documentation & Reproduction

- Library & Documentation

1977 was a year of re-orientation with relation to the needs of the new programmes of the Petten Establishment. With the assistance of a newly formed library-committee a start has been made with the following tasks:

- set-up of a new reference-section
- introduction of the UDC-system
- installing of archives
- determination of non-relevant literature, followed by storage
- planning of a new library-arrangement.

Normal routine-work also continued. The book collection, the periodical collection and the report collection increased to 6700 items, 2780 volumes and 9300 reports respectively. The documentation service treated 1700 literature-requests.

- Reproduction

A new reporting-system with new guidelines for the JRC was introduced. This resulted in an increase of the number of publications.
The reproduction-unit provided for 51 reports and 29 external reports/publications (see part IV of this report), placed 90 orders to external printers, and executed 80 smaller and 8 large lay-out orders.
The photographic laboratory carried out routine-work on neutrography, printed circuit mask production and etching as well as scientific and general photography and preparation of screens for reports.

5.2 Theoretical Analysis (Computer Services)

The PDP-15 system has been enlarged by the addition of a PDP-11 multi-user system with 96 K byte memory, 10 Megabyte disc and a dual floppy-disk drive: the whole being controlled by three console terminals (see Fig. 1). This tandem system combines the access convenience of the PDP-11 system with the capacity of the PDP-15 and is able to handle simultaneous data processing and calculation tasks.
During the year data from a large number of BWFC experiments in the HFR have been processed using MACRO code, and heat transfer calculations for two HTR fuel element capsules and for specially instrumented LWR fuel irradiation experiments were performed,

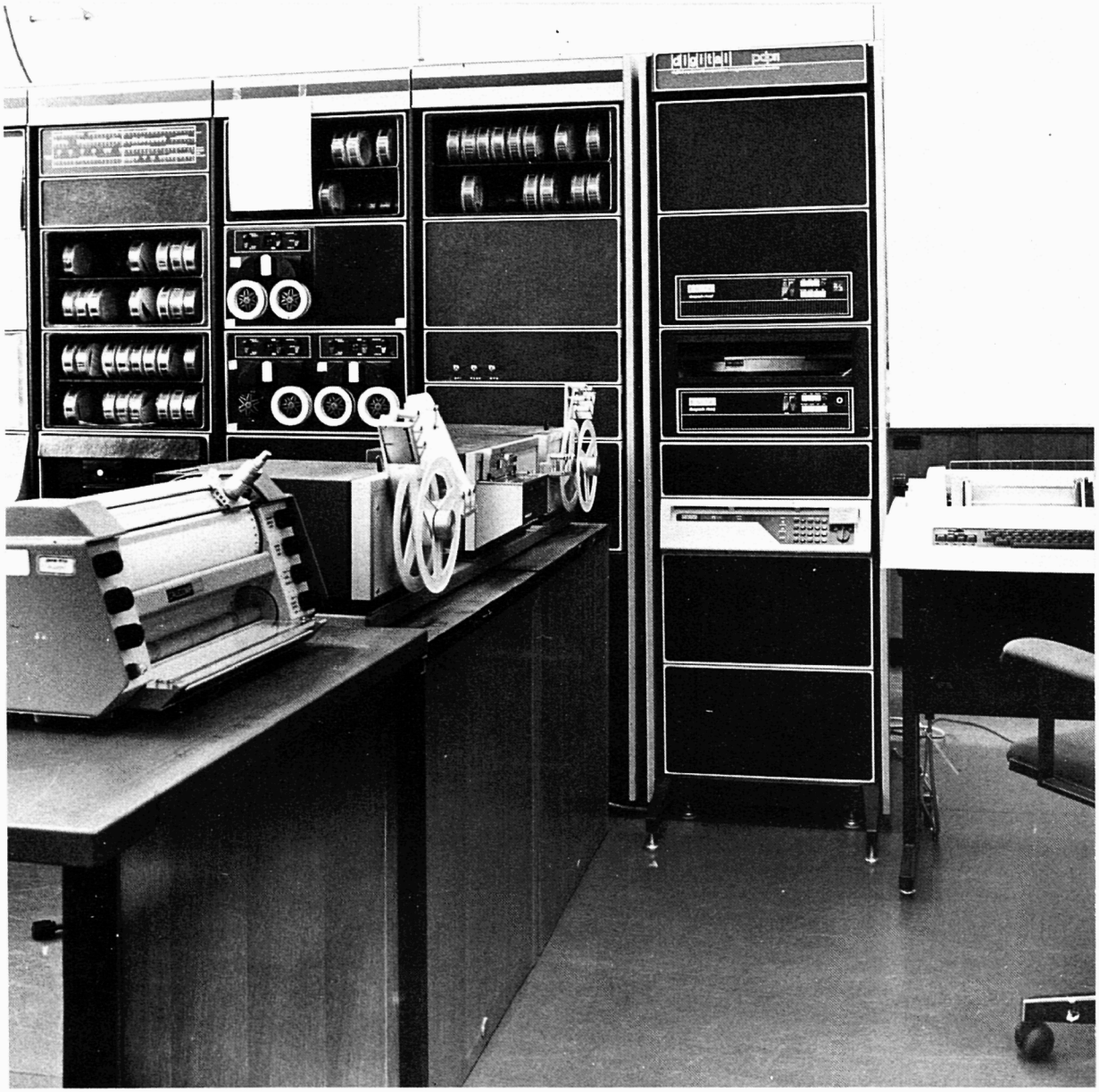


Fig. 1 A corner of the PDP-15/PDP-11 computer room.

where special analytical problems prevented the use of routine programmes.
For the Materials Division, general programmes have been developed to calculated gaseous compositions to provide equilibrated corrosive atmospheres with known component concentrations at various temperatures and pressures. Hydrogen, carbon, oxygen, sulphur and nitrogen have been considered.

5.3 Drawing Office and Workshops

During the year a series of new LMFBR fuel irradiation capsules have been designed and remote encapsulation equipment including in-cell NaK filling and welding conceived for the HFR programme.
The workshops have built an extensive panel system (Fig. 2) for the "Sweep Gas" experiment

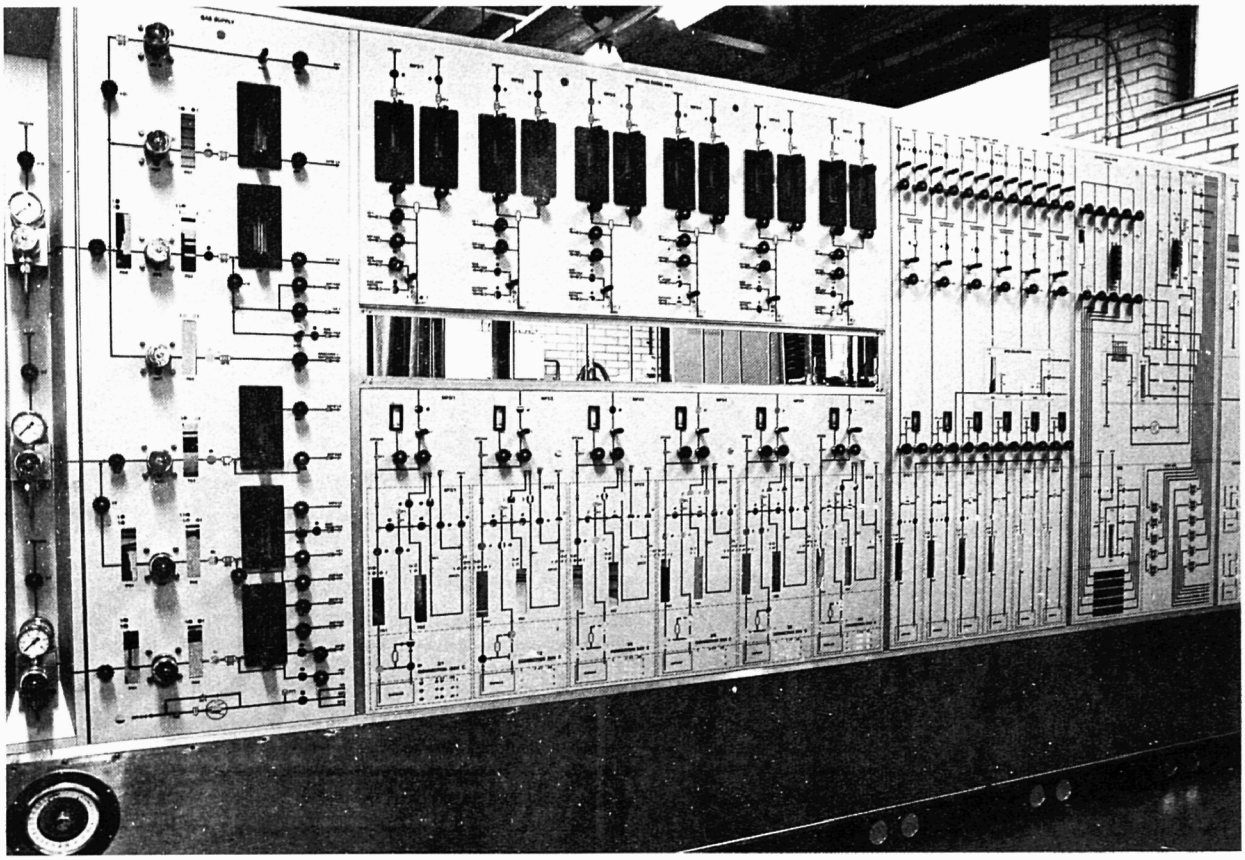


Fig. 2 "Sweep Gas" irradiation experiment. Command panel.

and developed spark erosion machining techniques for the accurate shaping of niobium drums and improved methods of ultra-precision machining of capsules tubes.
Work for the Materials Division has concentrated on the completion of an ion beam profile monitor (Fig. 3), employing a Nipkow disc to scan an ion beam emerging from a cyclotron, construction of furnaces and test loops for corrosion studies and a great deal of work to improve commercial mechanical test equipment. Notable has been the production of a large number of tensile test specimens to high precision, often in metals and alloys presenting difficulties due to hardness, toughness, etc.
Work has also been carried out on the production and modification of analytical equipment for the organic chemistry group.
The following table gives the effort in man years devoted to the two scientific divisions :

Division	1975	1976	1977
HFR	39,3	39,2	37,7
Materials Research	2,8	3,3	4,0
Total	42,1	42,5	41,7

Of these totals, about 50°/o is subcontracted to manufacturing and design organisations.

The Establishment also possesses a small glass blowing workshop, attached to the Materials Division and work has been carried out for the High Temperature Materials Programme, partly machining of ceramics, and partly glasswok, but principally for the Organic Materials Laboratories. Apart from general glass blowing, chromatographic columns in the form of long, continuous spirals of quartz of glass capillary tubing were required for their investigations. A commercial machine was succesfully modified to prepare these to constant dimensions.

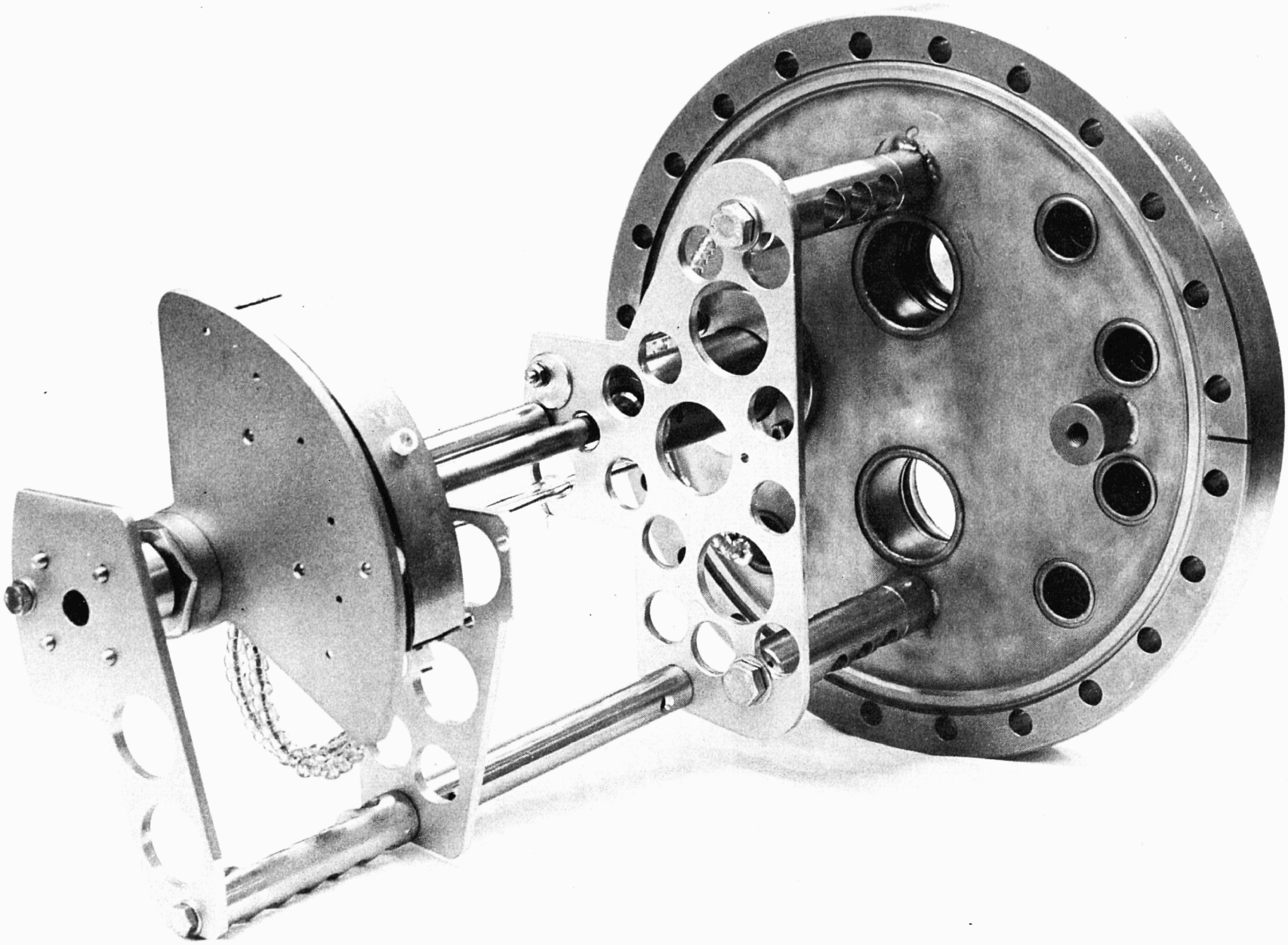


Fig. 3 Beam profile monitor sampling head, showing disc.

5.4 Electronics and Instrumentation

During 1977 the group has carried out work in support of both the HFR and Materials Divisional activities with HFR activities absorbing about 90 °/o of the available effort. An evolution has taken place in the work for the HFR projects where the development and manufacture of large instrumentation systems, and their maintenance is replacing the traditional "on bench" experimental and developmental activities. Staff formerly engaged in conventional electronic instrumentation work have become more involved in systems analysis and specification for production by sub-contract, project chasing, and software for on-line computer control and data handling.

The following important projects have been tackled for HFR experiments:

- The BWFC capsule control system; initially equipped for four irradiation capsules, it has operated reliably during the year. A second system for four more capsules has been provided and plans developed to use a PDP11/04 computer for on-line data analysis.
- COSAC; data handling system utilising an on-line PDP11.
- Sweep Gas Project; large scale instrumentation for this project contains a number of units as shown in Fig. 4. Notable is the use of a programmable logic controller (PLC), a Siemens Simatic S31 to control the experiment.

In addition, instrumentation for a number of smaller systems has been provided. Notable is a "state of the art" transducer amplifier.

For the Materials Laboratory work has been largely devoted to system development and specification for the Environmental Test Facility.
Smaller scale instrumentation has involved:

- design of safety control instrumentation for a corrosion rig operating with toxic and explosive atmospheres. A Simatic S31 Programmable Logic Controller (PLC) has been employed.
- realisation of a logarithmic scaling unit for use with a Linseis recorder for the physical metallurgy section, which operates by advancing the paper chart so as to create a logarithmic time scale; time markers are provided, (see Figs. 5 and 6).

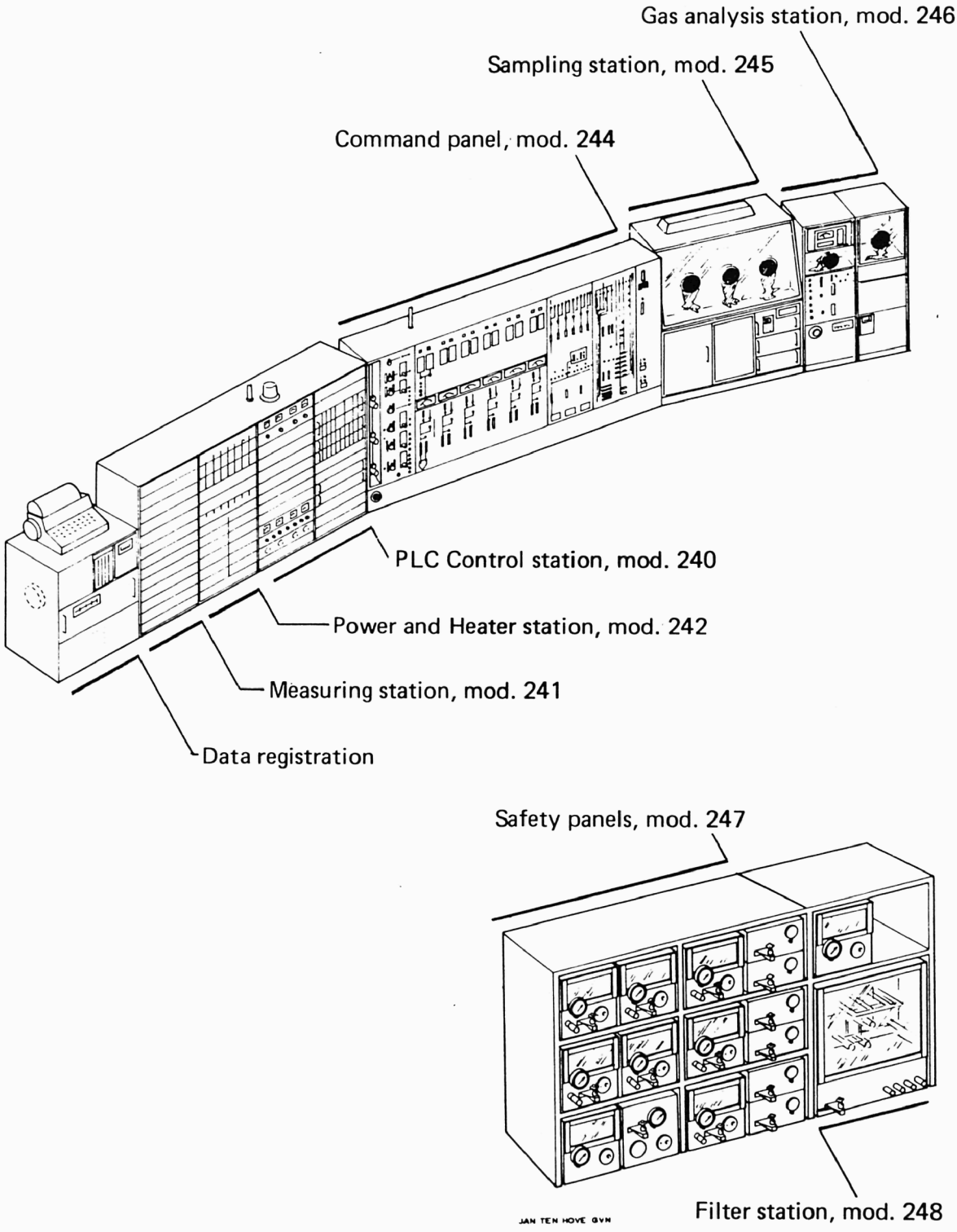


Fig. 4 Sweep gas instrumentation.

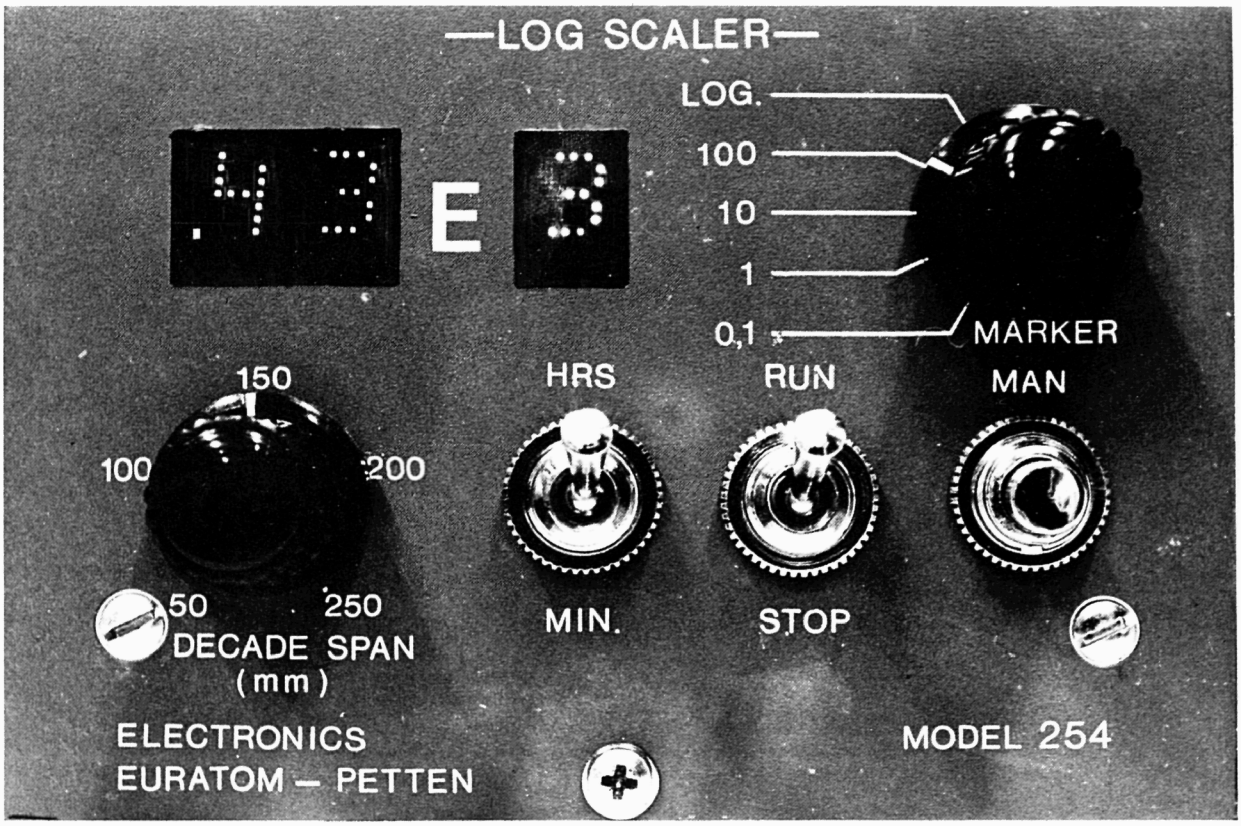


Fig. 5 Log scaler, front view.

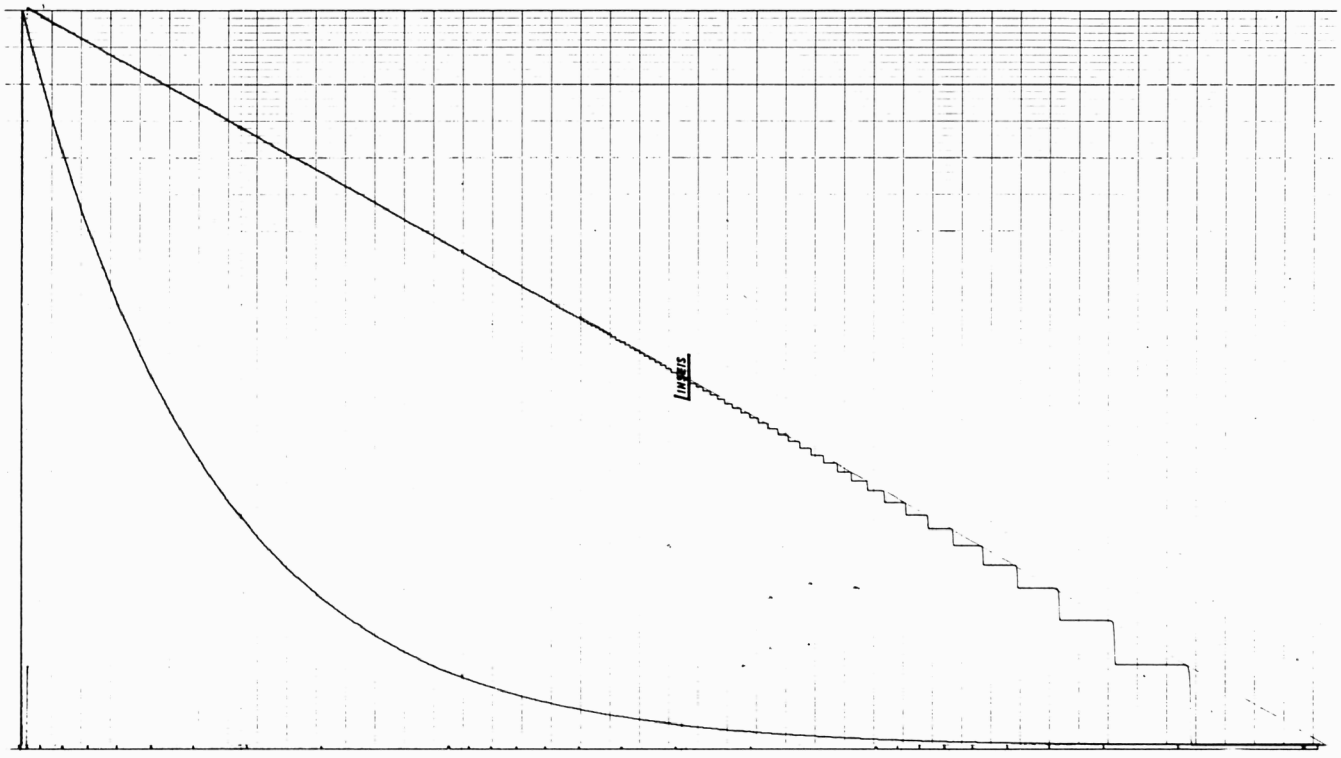


Fig. 6 Log scaler, performance: lower trace - output of a counter-DAC chain, upper trace - same signal passed through a logarithmic amplifier.

IV SCIENTIFIC PUBLICATIONS

SCIENTIFIC PUBLICATIONS

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Journal of Physics E (Scientific Instruments), Vol. 11 (1977), p. 333 - 335.

Bressers, J.; Cambini, M.:
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Journal of Nuclear Materials 68 (1977), p. 250-252.

Bressers, J.; Cassanelli, G.; De Cat, R.; Kohnen, H.; Gherardi, H.
Ultra-high vacuum target assembly for charged particle irradiations in the materials research field.
EUR 5908 e (October 1977)

Bressers, J.; Cambini, M.:
Irradiation damage in vanadium.
Contribution to "HFR Users' Meeting", 25 - 26 October 1977, at Petten.

Bressers, J.:
Study of deformation rate controlling mechanisms in vanadium at temperatures $T \leqslant 0.15\ T_m$.
EUR 5980 e, (to be published in 1978)

Cammarata, V.:
Power modulation testing of pre-irradiated fuel pins.
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Cundy, M.R.; Lölgen, R.H.; Schüle, W.:
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Post-irradiation examination of an 1300 ^{o}C HTR fuel experiment.
EUR 5841 e (July 1977).

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Lecture held at "Symposium de Spectrometrie de Masse", 20 - 21 April 1977 at Ruell (France).

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The damage to activation ratio for graphite irradiation in the HFR.
EUR 5795 e (June 1977).

Helbach, P.:
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Lölgen, R.; Everett, M.R.; Pott, G.; Thomson, J.M.;
Joint programme of uni-axial creep measurements of HTR compacts and matrix material under tensile load at 900 ^{o}C.
J. of Nuclear Materials, 65 (1977), p. 107-115.

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EUR 5463 e. Part 2.

Röttger, H. (ed.):
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EUR 5667 ef, supplement (August 1977).

Röttger, H.; Tas, A.; Van de Werve, H.; Von der Hardt, P.; Zijp, W.L.:
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EUR 5700 e REV.

Ruyter, I.; Markgraf, J.; Vogl, W.:
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Modification of the microstructure of IN100 by simulated coating heat treatments.
EUR 5887 e.

Van de Voorde, M.H.:
Programme Progress Report (PPR) - high temperature materials - first semester 1977. Summary and Main report.
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Final report on the 1975/76 programme: High temperature materials.
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Electronic Engineering, Vol. 49 (1977), no. 600, p. 35.

Veringa, H.J., Cundy, M.R.:
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Lecture held at "13th Carbon Conference", Irvine Cal., USA, 17-22 July 1977.
An abstract is to be published in Carbon.

Von der Hardt, P.:
High flux materials testing reactor (HFR) Petten. Progress report, January-June 1977.
COM 3431.

Von der Hardt, P.
Final report on the 1973-1976 programme.
to be published as communication.

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Annual report 1976.
EUR 5764 e (February 1977).

Materials Division J.R.C. Petten. Facilities and Equipment.

Reactor Radiation Metrology Newsletter.
(Ed.: H. Röttger, W. Schneider, W.L. Zijp)
No. 4, March 1977, No. 5, June 1977, No. 6, September 1977.

Proceedings of the HFR Users' Meeting, 25-26 October 1977, Petten.

V

INTERNATIONAL CONFERENCES

INTERNATIONAL CONFERENCES :

Survey of international conferences, attended by members of the JRC Petten staff in 1977.

"The Directive and the Cosmetic Industry"	London	17 February
"Utilisation of fossile fuels and associated corrosion problems in energy conversion systems"	Newcastle-u.-Tyne	15 March
"Sondermetalle"	Essen	16 March
"Transducer '77 Conference"	Tavistock	15 - 17 March
'Synthetic fuels from coal"	Eindhoven	24 March
"Gelpermeatiechromatographie	Etten-Leur	25 March
"Reaktortagung 1977"	Mannheim	29 March - 1 April
"Symposium de Spectrométrie de masse"	Paris	20 - 21 April
"International Conference on Nuclear Power and its Fuel Cycle"	Salzburg	2 - 8 May
"Journées de calorimétrie et d'analyse thermique"	Paris-Orsay	11, 12, 13 May
"1977 High Temperature Corrosion Conference"	Düsseldorf	17 - 18 May
"International Symposium on Micro-mechanical Techniques"	Davos	22 - 27 May
"Seminar 'de re metallica' "	Reutte	23 -26 May
"Characterisation of Ultrasonic Equip-ment"	Ispra	3 June
"Inleiding in de vacuumtechniek"	Woerden	15 June
"4th SAC Conference	London	17 - 22 July
"13th American Carbon Conference"	Irvine	17 - 22 July

"International Conference on Solidification and Casting"	Sheffield	18 - 21 July
"Gordon Research Conferences: Corrosion"	New Hampshire	18 - 22 July
"SMIRT Conference"	San Francisco	15 - 19 August
"Decus Europe Symposium 1977"	London	6 - 8 September
"6th European Congress on Corrosion"	London	19 - 20 September
"Europäische Werkzeugmaschinen Ausstellung"	Hannover	20 - 21 September
"Schweissen und Schneiden Fachmesse"	Essen	22 - 28 September
"Monitoring toxic hazards seminar"	Sheffield	27 - 28 September
"Symposium on Polynuclear aromatic Hydrocarbons"	Columbus	28 - 30 September
"ASTM Conference on Chromatography"	San Francisco	2 - 5 October
"DKG- Jahrestagung 1977"	Timmerdorfer Strand	5 - 6 October
"INCO Power Conference 1977"	Lausanne	5 - 7 October
"Interkama 1977"	Düsseldorf	6 - 12 October
"International Meeting on Advanced LMFBR Fuels"	Tucson	9 - 13 October
"Fachtagung Tiefbohren"	Dortmund	10 - 11 October
"Materials for Coal Conversion"	Washington	11 - 13 October
"Prediction of Residual Lifetime of Constructions operating at High Temperatures"	Den Haag	3 - 4 November
"Symposium on Advances in Chromatography"	Amsterdam	7 - 10 November
"Structural Reliability"	Ispra	7 - 11 November
"Recent Developments on High Temperature Design Methods"	London	6 December

GLOSSARY

GLOSSARY

ACPM	Advisory Committee on Programme Management
AERE	Atomic Energy Research Establishment
AES	Auger Electron Spectroscopy
AISI	American Iron and Steel Institute
ARTEMIS	Amoeba Rig Test Experiment on kernel Migration. In-pile Simulation
ASTM	American Society for Testing and Materials
AUSTIN	Austenitic Steel Irradiation
BATAVIA	Bilateral Advanced Trio And Vented-capsule Irradiation Assessment
B.C.C.	Body Centred Cubic
BCR	Community Reference Bureau
BERO	Brandstof Element Rooster experiment
BEST	Brennelementsegment
BONI	Borosilicate glasses Neutron Irradiation
BR2	Belgian materials testing Reactor at Mol
BWFC	Boiling Water Fuel-element Capsule
CADO	Calorimetric Dosimetry
CARSON	Carbide fuel ultra-Sonic thermometer
CCT	Common Customs Tariff
CFRMF	Coupled Fast Reactivity Measurements Facility
CII	Central Information Index
CL	Confidence Limit
CLUSTER	Name of computer code
CNR	Consiglio Nazionale delle Richerche
CORROX	Corrosion experiment on Oxide fuels
COSAC	Computerized On-line Supervision of the data Acquisition
COVER	Name of computer code
CRIMP	Graphite in-pile creep machine
CRM	Certified Reference Material
CRYSTAL BALL	Computer programme to evaluate the neutron flux density spectrum
DAMSIG 77	Name of code data library
DAR	Damage to Activation Ratio
DC	Direct Current
DIN	Deutsche Industrie Norm
DISCREET	Discontinuous in-pile graphite creep testing
DOSCROSS 77	Name of code data library
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ECN	Energieonderzoek Centrum Nederland
EDN	Equivalent DIDO Nickel fast neutron fluence
EEC	European Economic Community
EN	European Norm
ENDF	Evaluated Nuclear Data File
ENEX	Environmental Expertise
ENREP	Environmental Research Projects
ESCA	Electron Spectrocopy for Chemical Analysis
EWGRD	Euratom Working Group on Reactor Dosimetry
FIT	Fissile Isotope Target
FOM	Institute for Fundamental Materials Research Jutphaas, the Netherlands
FP	Flash Point
GC	Gas Chromatography
GFK	Gesellschaft für Kernforschung (Karlsruhe)
GIF	Gamma Irradiation Facility
GLC	Liquid Gas Chromatography
GPC	Gel Permeation Chromatography
GUD	Gestion d'Union Douanière
HB	Horizontal Beamhole
HFPIF	High Flux Poolside Isotope Facility
HFR	High Flux Reactor

HIFI	High Flux Facility for Isotopes
HPLC	High Performance Liquid Chromatography
HR	Hydraulic Rabbit Facility
HT	High Temperature
HTM	High Temperature Materials
HTR	High Temperature Reactor
HV	High Vacuum
IXAN	Commercial trademark (polymere)
IAEA	International Atomic Energy Agency
IP	Institute of Petroleum
JRC	Joint Research Centre
KFA	Kernforschungsanlage (Jülich)
LOC	Loss-of-cooling
LWR	Light Water Reactor
MACRO	Computer programme for data acquisition
METRE	Measurements, Standards and Reference Techniques
MRC	Metal Research Corporation
MS	Mass Spectrometry
MTR	Materials Testing Reactor
NAST	Natrium-Stahl
NBS	National Bureau of Standards
NIRVANA	Niobium and Vanadium irradiation in Na (sodium)
PAH	Polyaromatic hydrocarbons
PDP	Trademark for "Digital Equipment Corporation" computers
PIF	Poolside Isotope Facility
POCY	Power Cycling experiment
PRM	Reference materials with certified physical and/or technical properties
PROF	Poolside Rotating Facility
PRS	Pneumatic Rabbit System
PSF	Poolside Facility
R & D	Research and Development
REFA	Reloadable Facility
RFSP-JÜL	Computer programmes to evaluate the neutron flux density spectrum
RIF	Reloadable Isotope Facility
RM	Reference Material
SAND-II	Spectrum Analysis by Neutron Detectors
SANDAMAGE	Name of code data library
SEM	Scanning Electron Microscopy
SHOT	Stationary High Overtemperature
SINAS	Simplified NAS(T)
SNR	Schneller Natriumgekühlter Reaktor (Kalkar)
SP	Softening Point
STEK	Snel Thermisch Experiment Krito
STEM	Scanning Transmission Electron Microscope
TEDDI	Computer programme to evaluate reactor neutron spectrum
TEM	Transmission Electron Microscope
TLC	Thin Layer Chromatography
TOP	Transient Overpower experiment
TRESON	Mesure de Transport d'Energie en pile par méthodes Soniques
TRIO	Irradiation device with three thimbles
UA	Unit of Account
USNRS	Nuclear Regulatory Commission (USA)
UV	Ultraviolet
WFIRO	computer programme for two-dimensional heat transfer (r,o)
WOL	Steel specimen code
WRZRO	Computer programme for two-dimensional heat transfer (r,z)
WSG	Working Subgroup
WUNRO	Computer programme for one-dimensional heat transfer (r)
XPS	X-ray Photoelectron Spectroscopy

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